PRC'D 6-19-92 F.B. NAME: Ciba Gergy
1.D. NO.: RIDOO 1198323
FILE LOCIRA
OTHER: Oversize

CHANGES TO THE RCRA FACILITY INVESTIGATION

CIBA-GEIGY FACILITY CRANSTON, RHODE ISLAND

QUALITY ASSURANCE DOCUMENTS: SUPPLEMENT PARTS 1 AND 2

Submitted by: CIBA-GEIGY CORPORATION 444 Saw Mill River Road Ardsley, NY 10502

June 1992



NAME: CLEA BERRY
I.D. NO: REDOUTIGY3723
FILE 100R9
OTHER: Oversize

CHANGES TO THE RCRA FACILITY INVESTIGATION

CIBA-GEIGY FACILITY CRANSTON, RHODE ISLAND

QUALITY ASSURANCE DOCUMENTS: SUPPLEMENT PARTS 1 AND 2

Submitted by: CIBA-GEIGY CORPORATION 444 Saw Mill River Road Ardsley, NY 10502

June 1992

CRANSTON QAPJP TRACKING FORMS

CRANSTON OAP TRACKING FORM

No. 1 Date: 05/15/92

PERSON REQUESTING REVISION: Diana Baldi

RESPONSIBILITY OF REQUESTOR IN CRANSTON PROJECT: CIBA-GEIGY Corporation National Service Contract Administrator

NATURE OF CHANGE TO QAPIP:

Type: 1. Major___ Minor_X (check one)

2. Informational X Technical (check one)

<u>Section(s)</u>: 2, 3, and 17

USEPA Project Manager

of Document: CIBA-GEIGY QA Documents Supplement #1

REVISION: (1) Items K, L, and M added to List of Appendices on pages 2-5 and 3-4. (2) Following names/organizations added to Section 2.4 Distribution list and Section 17: Steve Bishop, Savannah Analytical Services; Jerry Zschau, R.I. Analytical Laboratories, Inc.; Robert E. Wagner, Northeast Analytical, Inc. (3) Telephone numbers of Diana Baldi and Tom Barber corrected in Section 2, Table of Contents.

<u>REASON FOR CHANGE</u>: (For Items 1 & 2) Three additional laboratories selected to analyze environmental samples to support the river modeling. (For Item 3) Correction of telephone numbers.

<u>EFFECTS OF CHANGE</u>: Requested detection limits for modeling and assistance with ISCO bottle preparation will be obtained. Telephone numbers are current.

NECESSARY CORRECTIVE ACTION(S): (1) Immediate project-wide distribution of additional quality assurance documents submitted by participating laboratories (Appendices K, L, and M) and Sections 2, 3, 17 and 18 (Change Form #1) of the CIBA-GEIGY QA Documents Supplement #1 is needed. (2) Additional informational corrections are needed in the CIBA-GEIGY QA Documents Supplement #1, Sections 4 (pages 4-2 and 4-11) and Section 9 (page 9-1). The implementation of the changes in Sections 4 and 9 will be made when the first revisions are made to these sections based on a technical change. Note that technical changes to these sections are expected within the next 90 days.

Jiana Galdi Project Manager	<i>5/26/92</i> Date
Responsibility: National Service Contract Administrator	
CIBA-GEIGY Project Coordinator	5/29/92
CIBA-GEIGY Project Coordinator	Dáte /
APPROVED (if technical change):	

Date

CRANSTON QAPJP TRACKING FORM

No. 2 Date: 05/26/92

PERSON REQUESTING REVISION: Thomas Barber

RESPONSIBILITY OF REQUESTOR IN CRANSTON PROJECT:
Group Leader, Analytical Chemistry, CIBA-GEIGY Corporation

NATURE OF CHANGE TO QAPIP:

Type: 1. Major___ Minor_X (check one)

2. Informational ____ Technical X (check one)

Section(s): 2 and Appendices C and D

of Document: CIBA-GEIGY QA Documents Supplement # 1 - APPENDIX H

REVISIONS: (1) Addition of two Standard Operating Procedures (SOPs) for the quantitative determination of silver and zinc by Zeeman Graphite Atomic Absorption using low concentration digestion techniques. (2) Addition of Alan Lynch, Inorganics Laboratory Supervisor, to CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan distribution list.

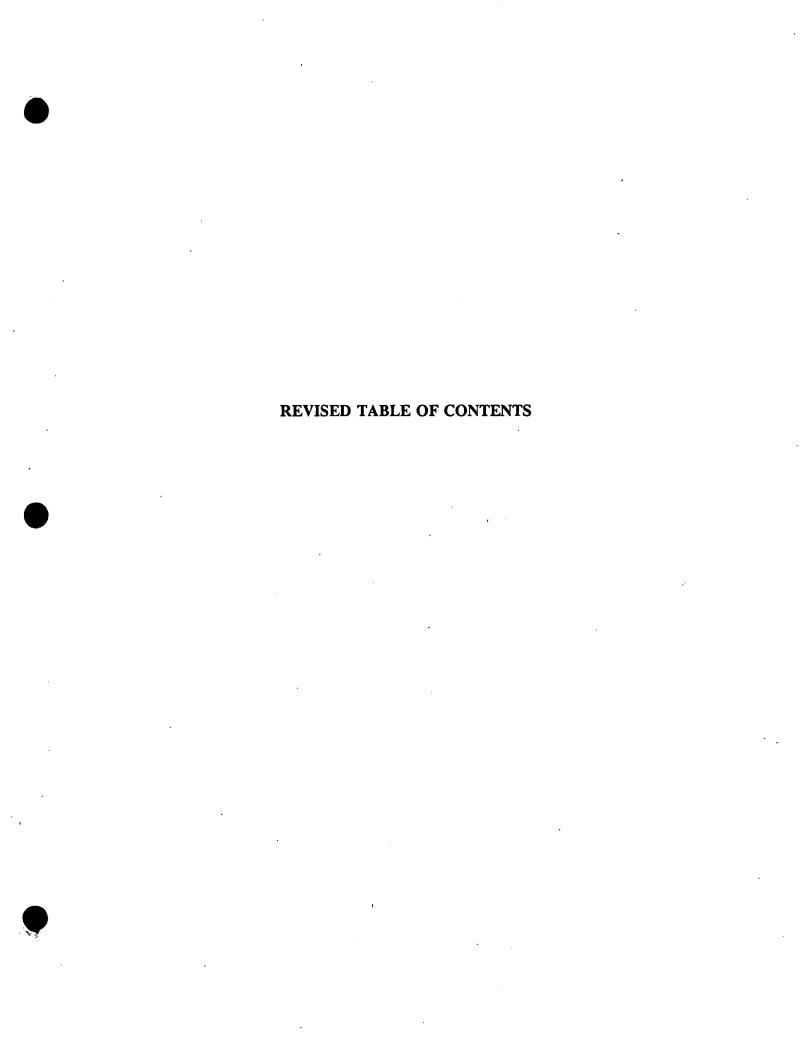
REASON FOR CHANGE: (1) Low detection limits are required for Pawtuxet River sampling program. (2) Additional personnel are involved in the project.

EFFECTS OF CHANGE: Addenda to CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan (Appendices C and D)

NECESSARY CORRECTIVE ACTION(S): Project-wide distribution of Section 18 (Change Form #2) with following attachments pertaining to Appendix H of CIBA-GEIGY Quality Assurance Documents - Supplement #1:

- (1) Section 2 of CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan
- (2) Appendices C and D of CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan

Dhana Balde	5/26/92
Project Manager Responsibility: National Service Contract Administrator	Date '
CIBA-GEIGY Project Coordinator	<u>5/29/92</u> Date
APPROVED (if technical change):	
USEPA Project Manager	Date



Section No.: 2
Revision No.: 1
Date: 5/15/92
Page 1 of 8

2.0 TABLE OF CONTENTS

		Revision No.	No. of <u>Pages</u>
1.0	TITLE PAGE	0	1
2.0	TABLE OF CONTENTS	1	8
	2.1 List of Figures2.2 List of Tables2.3 List of Appendices2.4 Distribution List		
3.0	PROJECT DESCRIPTION	1	3
	3.1 Project History3.2 Site Location and Description3.3 Intended Data Uses3.4 Organization of This Document		·
4.0	PROJECT ORGANIZATION AND RESPONSI-BILITIES	0	11
	4.1 Rationale for Project Organization4.2 Project Organization4.3 Responsibilities		
5.0	QUALITY ASSURANCE OBJECTIVES	0	18
	5.1 Overview 5.2 PARCC Parameters 5.2.1 Precision 5.2.2 Accuracy 5.2.3 Representativeness 5.2.4 Comparability 5.2.4.1 Differences from the Appendix IX List 5.2.4.2 Differences from the pendix IX Recommende Methods and Practica Quantitation Limits 5.2.5 Completeness	Ap-	
6.0	SAMPLING PROCEDURES	0	1
7.0	SAMPLE CUSTODY	. 0	1
8.0	CALIBRATION PROCEDURES AND FREQUENCY	0	1

Section No.: 2
Revision No.: 1
Date: 5/15/92
Page 2 of 8

			Revision	
9.0	ANALY	TICAL PROCEDURES	0	1
10.0	DATA	REDUCTION, VALIDATION AND REPORTING	0	13
	10.3	Objectives of Data Management Data Not Maintained in the Project Data Base Data Maintained in the Project Data Base 10.4.1 Field Information in the Project Data Base 10.4.2 Laboratory Results in the Project Data Base 10.4.3 Validation of Key Fields During Data Upload 10.4.4 Raw, Validated, and Final Data Loading the Raw Data Validating the Data		
11.0	INTER	NAL QUALITY CONTROL CHECKS	0	7
	11.2	Overview Field Quality Control Laboratory Analysis Quality Control Data Management Quality Control 11.4.1 System Security		
12.0	PERFO	RMANCE AND SYSTEM AUDITS	0	2
3	12.2	Overview System Audits Performance Audits		·
13.0	PREVE	NTIVE MAINTENANCE	0	1
		Overview Data Base Preventive Maintenance		

Section No.: 2 Revision No.: 1 Date: 5/15/92 Page 3 of 8

			Revision No.	
14.0		PROCEDURES USED TO ASSESS DATA N, ACCURACY, REPRESENTATIVENESS		
	AND COMP		0	1
15.0	PROCEDUR	ES FOR CORRECTING DEFICIENCIES	0	1
16.0	QUALITY .	ASSURANCE REPORTS TO MANAGEMENT	0	3
		erview tification to EPA visions to Document		
17.0	CURRENT	PERSONNEL FOR KEY POSITIONS	1	3
18.0		CHANGES TO THIS QUALITY ASSURANCE DO PPLEMENT #1	0 0	1
2.1	LIST OF F	IGURES		
Fig	ure 4-1	Organizational Chart for Cranston Project (titles only)		
Fig	ure 10-1	Data Flow for Analytical Samples		
Fig	ure 16-1	Revision to QAPjP Tracking Form		
Fig	ure 17-1	Organizational Chart for Cranston	Project	
2.2	LIST OF T	ABLES		
Tab	ole 4-1	Constituents Analyzed for Cranston Listed by Category of Analysis	Project	
Tab	ole 4-2	Identification and Status of Selector Laboratory Analysis	ted Vendor	·s
Tab	ole 5-1	Target Percent Recovery (%R) and Re Difference (RPD) Limits for Matrix and Laboratory Control Samples for	Spike Sam	ples
Tab	ole 5-2	Target Percent Recovery (%R) and Re Difference (RPD) Limits for Matrix and Laboratory Control Samples for Compounds and Analytes	Spike Sam	

Section No.: 2 Revision No.: 1 Date: 5/15/92 Page 4 of 8

2.2 LIST OF TABLES (cont.)

- Table 5-3 Target Percent Recovery (%R) and Relative Percent Difference (RPD) Limits for Matrix Spike Samples for Inorganic Parameters
- Table 5-4 Target Percent Recovery (%R) and Relative Percent Difference (RPD) Limits for Matrix Spike Samples for Laboratory Control Samples (LCS) for Inorganic Parameters
- Table 5-5 Target Accuracy Acceptance Limits for Percent Recovery of Surrogate Compounds
- Table 5-6 List of Compounds/Analytes Analyzed by Savannah Laboratories, Inc. (SLI) Using a Method which Differs from the Recommended Method in the Appendix IX List
- Table 5-7 List of Compounds/Analytes Analyzed by PACE, Inc., Using a Method which Differs from the Recommended Method in the Appendix IX List
- Table 5-8 List of Compounds that SLI reports a higher PQL than Both the Recommended Appendix IX PQL and the PQL Generated Historically for the Project by Radian
- Table 5-9 List of Compounds that PACE Reports a Higher PQL than Both the Recommended Appendix IX PQL and the PQL Generated Historically for the Project by Radian
- Table 10-1 Fields in the Sample Collection Table
- Table 10-2 Fields in the Sample Analysis Table
- Table 11-1 Definitions of Data Qualifiers for All Qualifier Fields
- Table 11-2 Rules for Translating the Data Qualifiers During the Load Procedure from the Raw Data Qualifier Field to the Validated Data Qualifier Field
- Table 11-3 Valid Data Qualifiers for the First Two Stages of Data Upload and Validator Editing
- Table 12-1 System Audits for Cranston Project

Section No.: 2 Revision No.: 1 Date: 5/15/92 Page 5 of 8

2.2 LIST OF TABLES (cont.)

Table 17-1 List of Current Personnel in Key Positions

Table A-1 Record Layout for FRACTION.PRN File for Data on Diskette

DISKecce

Table A-2 Record Layout for RESULTS.PRN File for Data on

Diskette

2.3 LIST OF APPENDICES

A. CIBA-GEIGY Database Management Detail

B. Woodward-Clyde Data Collection Quality Assurance Project Plan (QAPjP)

C. Savannah Laboratories, Inc., QAPjP

D. PACE, Inc., QAPjP

E. Triangle Labs of Houston QAPjP

F. Enseco-CAL QAPjP

G. CIBA-GEIGY Environmental Testing Lab (ETL) QAPjP

H. CIBA-GEIGY Analytical Chemistry QAPjP

I. IT Corporation QAPjP for Biological Fieldwork and TIE

J. IT Corporation QAPjP for Geotechnical Analyses

K. Savannah Analytical Services Quality Assurance Document

L. R.I. Analytical Laboratories, Inc., Quality Assurance Document

M. Northeast Analytical, Inc., Quality Assurance Document

2.4 DISTRIBUTION LIST

ENTIRE DOCUMENT:

Frank Battaglia, USEPA Region I (6 copies)

Waste Management Building

90 Canal Street

Boston, MA 02114

Voice: (617) 573-9643

FAX: (617) 573-9662

Diane Leber, CIBA-GEIGY (x2159); FAX: (914) 479-2332

Corporate Environmental Protection

Patty Culver, CIBA-GEIGY (x4365); FAX: (914) 479-2290

Corporate Information Services

CIBA-GEIGY Corporation

444 Saw Mill River Road

Ardsley, NY 10502

Voice: (914) 479-5000

Section No.: 2 Revision No.: 1 Date: 5/15/92 Page 6 of 8

ENTIRE DOCUMENT (cont.):

Diana Baldi, CIBA-GEIGY
CIBA-GEIGY Corporation
Environmental Technology Center
P. O. Box 18300 (27419), 410 Swing Road (27409)
Greensboro, NC
Voice: (919) 632-7506
FAX: (919) 632-2048

Frank Saksa, CIBA-GEIGY (x2789)
CIBA-GEIGY Corporation
Environmental Testing Laboratory
P. O. Box 71, Route 37 West
Toms River, NJ 08754
Voice: (908) 349-5200
FAX: (908) 505-9195

Mark Houlday, Woodward-Clyde Consultants 201 Willowbrook Boulevard P. O. Box 290 Wayne, NJ 07470 Voice: (201) 785-0700 FAX: (201) 785-0023

Tom C. Marshall, IT Corporation 312 Director's Drive Knoxville, TN 37923 Voice: (615) 690-3211 FAX: (615) 690-3626

John Connolly, HydroQual, Inc. 1 Lethbridge Plaza Mahwah, NJ 07430 Voice: (201) 529-5151 FAX: (201) 529-5728

Section No.: 2 Revision No.: 1 Date: 5/15/92 Page 7 of 8

MAIN DOCUMENT WITH APPENDICES A AND B:

Linda Wolfe, Savannah Laboratories, Inc. 5102 La Roche Avenue (31404)
P. O. Box 13548 (31416-0548)
Savannah, GA
Voice: (912) 354-7858
FAX: (912) 352-0165

Bill Davis, PACE, Inc.
Robinson Lane, RD6
Wappingers Falls, NY 12590
Voice: (914) 227-2811
FAX: (914) 227-6134

Rod Miller, PACE, Inc. 1710 Douglas Drive North Minneapolis, MN 55422 Voice: (612) 544-5543 FAX: (612) 525-3377

Ed Stuber, PACE, Inc.
Robinson Lane, RD6
Wappingers Falls, NY 12590
Voice: (914) 227-2811
FAX: (914) 227-6134

Diane Waldschmidt, PACE, Inc. 100 Marshall Drive Warrendale, PA 15086 Voice: (412) 772-0610 FAX: (412) 772-4020

Shelly Eyraud, Enseco-CAL 2544 Industrial Boulevard West Sacramento, CA 95691 Voice: (916) 372-1393 FAX: (916) 372-1059

Steve Missler, Triangle Labs of Houston 12823 Park One Drive Sugarland, TX 77478 Voice: (713) 240-5330 FAX: (713) 240-5341

Section No.: 2 Revision No.: 1 Date: 5/15/92 Page 8 of 8

MAIN DOCUMENT WITH APPENDICES A AND B: (cont.)

John Rissel, CIBA-GEIGY (x2262)
Environmental Testing Laboratory
P. O. Box 71, Route 37 West
Toms River, NJ 08754
Voice: (908) 349-5200
FAX: (908) 505-9195

Tom Barber, CIBA-GEIGY
Environmental Technology Center
P. O. Box 18300 (27419), 410 Swing Road (27409)
Greensboro, NC
Voice: (919) 632-7297
FAX: (919) 632-2048

Dan Duh, IT Corporation 165 Fieldcrest Avenue Edison, NJ 08837 Voice: (908) 225-2000 FAX: (908) 225-1691

FAX: (615) 482-1890

Tom Geisler, IT Corporation
Non-U.S. Mail: 1570 Bear Creek Road
Oakridge, TN 37830
U.S. Mail: 1570 Bear Creek Road
Kingston, TN 37763
Voice: (615) 482-6497

Steve Bishop, Savannah Analytical Services
Suite "I"
42 West Montgomery Crossroads
Savannah, GA 31406
Voice: (912) 920-4725
FAX: (912) 927-2076

Jerry Zschau, R.I. Analytical Laboratories, Inc. 41 Illinois Avenue Warwick, RI 02888 Voice: (401) 737-8500 FAX: (401) 738-1970

Robert E. Wagner, Northeast Analytical, Inc. 301 Nott Street Schenectady, NY 12305 Voice: (518) 346-4592 FAX: (518) 381-6055

REVISED SECTION 3

Section No.: 3
Revision No.: 1
Date: 05/15/92
Page 1 of 4

3.0 PROJECT DESCRIPTION

3.1 Project History

A RCRA Corrective Action Study has been undertaken by CIBA-GEIGY at its former manufacturing facility in Cranston, Rhode Island, pursuant to a Consent Agreement and Order (No. 1-88-1088) entered into by CIBA-GEIGY Corporation and the United States Environmental Protection Agency (USEPA), effective 16 June 1989. The RCRA Corrective Action Study has four stages:

Stage 1: RCRA Facility Assessment. In 1987, the USEPA conducted the RCRA Facility Assessment (hereafter called the "Facility Assessment") to identify known and/or suspected releases at the facility requiring further action. The results were presented in the Final RFA Report, CIBA-GEIGY RCRA Facility Assessment (January 1988). In 1988, CIBA-GEIGY conducted a Preliminary Investigation (not required by the Order) to begin characterizing the facility's environment and selected releases. The results of the Preliminary Investigation were summarized in Chapter 1 of the Current Assessment Summary Report (1990).

Stage 2: RCRA Facility Investigation. The RCRA Facility Investigation (hereafter simply called the "Facility Investigation") will characterize the nature and extent of any known or suspected releases from Solid Waste Management Units (SWMUs) and Areas of Concern (AOCs) that were determined by the Facility Assessment to require further action. The Facility Investigation is being conducted in two phases; CIBA-GEIGY proposed that Phase I be conducted in two parts -- Phases IA and IB -- to obtain additional guidance from the USEPA throughout the project. Phase IA was conducted in late 1989 and mid-1990 to characterize the facility's physical environment more completely; the results of the Phase IA studies were presented in the Phase IA Report (approved in June 1991). Phase IB was conducted in late 1990 and early 1991; it characterized known and/or suspected releases of the facility more completely and also provided additional information about the facility's physical environment. Sampling in

Section No.: 3
Revision No.: 1
Date: 05/15/92
Page 2 of 4

Phase IB was conducted in two rounds. The <u>Phase I Interim Report</u> (submitted in November, 1991) presents the results of both Phases IA and IB. Phase II, scheduled to begin after the USEPA approves the <u>Phase II Proposal</u>, will entail additional site characterization and sampling, the Public Health and Environmental Risk Evaluation (PHERE), and the proposal of Media Protection Standards.

Stage 3: Corrective Measures Study Proposal. The Corrective Measures Study (CMS) Proposal describes the measures available to achieve the Media Protection Standards. Work on the CMS Proposal will begin after the USEPA approves the Media Protection Standards.

Stage 4: Corrective Measures Study Report. The Corrective Measures Study (CMS) Report evaluates the measures available to achieve the Media Protection Standards at the facility. Work on the CMS Report will begin after the USEPA approves the CMS Proposal.

3.2 Site Location and Description

The site is located in an urban setting in the communities of Cranston and Warwick, Rhode Island. The site adjoins residential areas to the north and south, a commercial area to the east, and open land (which was formerly the site of an industrial facility) to the west. The site slopes gently toward, and is divided by, the Pawtuxet River. The RCRA Corrective Action Study addresses three general areas:

- the on-site area (that is, the site itself);
- the off-site area (exclusive of the Pawtuxet River); and
- the Pawtuxet River.

In addition, the on-site area is divided into three study areas:

- the Production Area;
- the Waste Water Treatment Area; and
- the Warwick Area.

Within the on-site area, 12 SWMUs and two AOCs have been identified at the site. For completeness of the study, CIBA-

Section No.: 3
Revision No.: 1
Date: 05/15/92
Page 3 of 4

GEIGY identified two additional areas of investigation (AAOIs).

3.3 Intended Data Uses

The data collected during the multiple study phases will have various intended uses. Data collection objectives are described in detail in the workplan for each phase; however, in general the data have four main intended uses:

- 1. To identify compounds positively present in all required media of concern. In doing so, identify the nature and extent of contamination associated with SWMUs, AOCs, AAOIs, and required off-site areas, as apporopriate;
- 2. To provide representative data for assessing potential impacts to public health and the environment;
- 3. To evaluate remedial options for soil, sediment, and water; and
- 4. To provide pre-design data for selected remedial options.

3.4 Organization of This Document

This document describes the management system that will be used to ensure that data and information generated during the RCRA Corrective Action Study are technically sound and valid. Overall project quality is the responsibility of CIBA-GEIGY Corporation. In addition, each laboratory and technical consultant under contract to CIBA-GEIGY is responsible for data quality in their realm of expertise. Accordingly, this document was to function as an overall quality assurance management plan developed by CIBA-GEIGY. The appendices contain the Quality Assurance Documents developed by each laboratory and technical consultant performing work which generates data for use in this project. The following ten appendices are attached:

- A. CIBA-GEIGY Database Management Detail
- B. Woodward-Clyde Data Collection Quality Assurance Project Plan (QAPjP)

Section No.: 3
Revision No.: 1
Date: 05/15/92
Page 4 of 4

- C. Savannah Laboratories, Inc., QAPjP Chemical Analysis
- D. PACE, Inc., QAPjP Chemical Analysis
- E. Triangle Labs of Houston QAPjP Chemical Analysis for Dioxins and Furans
- F. Enseco-CAL QAPjP Chemical Analysis for Dioxins and Furans
- G. CIBA-GEIGY Environmental Testing Lab (ETL) QAPjP Chemical Analysis for Treatability Parameters
- H. CIBA-GEIGY Analytical Chemistry QAPjP PCB Screening
- I. IT Corporation QAPjP for Biological Fieldwork and TIE
- J. IT Corporation QAPjP for Geotechnical Analyses
- K. Savannah Analytical Services QAPjP for River Modeling Support
- L. R.I. Analytical Laboratories, Inc., QAPjP for River Modeling Support
- M. Northeast Analytical, Inc., QAPjP for River Modeling Support

REVISED SECTION 17

Section No.: 17
Revision No.: 1
Date: 05/15/92
Page 1 of 4

17.0 CURRENT PERSONNEL FOR KEY POSITIONS

Table 17-1 lists the current personnel for key positions, and Figure 17-1 shows the organizational structure for the Cranston Project.

TABLE 17-1 List of Current Personnel for Key Positions

AFFILIATION	TITLE	NAME	ADDRESS	TELEPHONE & FAX NO.
CIBA-GEIGY Corporation	Project Manager	Diane Leber	444 Saw Mill River Road Ardsley, NY 10502	(914)479-2159 FAX:(914)479-2332
CIBA-GEIGY Corporation	National Service Contract Administrator	Diana Baldi	410 Swing Road Greensboro, NC 27419	(919)632-7506 FAX:(919)632-2048
CIBA-GEIGY Corporation	Database Manager	Patty Culver	444 Saw Mill River Road Ardsley, NY 10502	(914)479-4365 FAX:(914)479-2290
CIBA-GEIGY Corporation	Quality Assurance Officer	Frank Saksa	Route 37 West Toms River, NJ 08754	(908)349-2789 FAX:(908)505-9195
CIBA-GEIGY Corporation	Lab Manager	John Rissel	Route 37 West Toms River, NJ 08754	(908)349-2262 FAX:(908)505-9195
CIBA-GEIGY Corporation	Group Leader	Tom Barber	410 Swing Road Greensboro, NC 27419	(919)632-7297 FAX:(919)632-2048

Section No.: 17 Revision No.: 1 Date: 05/15/92 Page 2 of 4

TABLE 17-1 (cont.) List of Current Personnel for Key Positions

AFFILIATION	TITLE	NAME	ADDRESS	TELEPHONE & FAX NO.
Woodward-Clyde Consultants	Project Manager	Mark Houlday	201 Willowbrook Blvd. Wayne, NJ 07470	(201)785-0700 FAX: (201)785-0023
HydroQual, Inc.		John Connolly	1 Lethbridge Plaza Mahwah, NJ 07430	(201)529-5151 FAX: (201)529-5728
IT Corporation	Project Manager	Thomas Marshall	312 Directors Drive Knoxville, TN 37923	(615)690-3211 FAX:(615)690-3626
IT Corporation	Lab Manager	Tom Geisler	1570 Bear Creek Road Kingston, TN 37763	(615)482-6497 FAX:(615)482-1890
IT Corporation	Lab Manager	Dan Duh	165 Fieldcrest Avenue Edison, NJ 08837	(908)225-2000 FAX: (908)225-1691
Savannah Labora- tories, Inc.	Project Manager	Linda Wolfe	5102 La Roche Avenue Savannah, GA 31404	(912)354-7858 FAX: (912)352-0165
PACE, Inc.	Project Manager	Bill Davis	Robinson Lane, RD6 Wappingers Falls, NY 12590	(914)227-2811 FAX: (914)227-6134
Enseco-CAL	Project Manager	Shelly Eyraud	2544 Industrial Blvd. West Sacramento, CA 95691	(916)372-1393 FAX: (916)372-1059
Triangle Labs of Houston	Project Manager	Steve Missler	12823 Park One Drive Sugarland, TX 77478	(713)240-5330 FAX: (713)240-5341

Section No.: 17 Revision No.: 1 Date: 05/15/92 Page 3 of 4

TABLE 17-1 (cont.) List of Current Personnel for Key Positions

AFFILIATION	TITLE	NAME	ADDRESS	TELEPHONE & FAX NO.
Savannah Analytical Serv.	Project Manager	Steve Bishop	Suite "I" 42 West Montgomery Crossroads Savannah, GA 31406	(912) 920-4725 FAX:(912) 927-2076
R.I. Analytical Laboratories, Inc.	Project Manager	Jerry Zschau	41 Illinois Avenue Warwick, RI 02888	(401) 737-8500 FAX:(401) 738-1970
Northeast Analytical, Inc.	Project Manager	Robert E. Wagner	301 Nott Street Schenectady, NY 12305	(518) 346-4592 FAX: (518) 381-6055

Section No.: 17 Revision No.: 1 Date: 05/15/92 Page 4 of 4

THIS PAGE RESERVED FOR FIGURE 17-1 PREPARED BY WOODWARD-CLYDE

REVISED SECTION 18

CRANSTON QAPIP TRACKING FORM

No. 1 Date: 05/15/92

PERSON REQUESTING REVISION: Diana Baldi

RESPONSIBILITY OF REQUESTOR IN CRANSTON PROJECT: CIBA-GEIGY Corporation National Service Contract Administrator

NATURE OF CHANGE TO QAPjP:

Type: 1. Major____ Minor_X (check one)

2. Informational X Technical (check one)

Section(s): 2, 3, and 17

of Document: CIBA-GEIGY QA Documents Supplement #1

REVISION: (1) Items K, L, and M added to List of Appendices on pages 2-5 and 3-4. (2) Following names/organizations added to Section 2.4 Distribution list and Section 17: Steve Bishop, Savannah Analytical Services; Jerry Zschau, R.I. Analytical Laboratories, Inc.; Robert E. Wagner, Northeast Analytical, Inc. (3) Telephone numbers of Diana Baldi and Tom Barber corrected in Section 2, Table of Contents.

<u>REASON FOR CHANGE</u>: (For Items 1 & 2) Three additional laboratories selected to analyze environmental samples to support the river modeling. (For Item 3) Correction of telephone numbers.

<u>EFFECTS OF CHANGE</u>: Requested detection limits for modeling and assistance with ISCO bottle preparation will be obtained. Telephone numbers are current.

NECESSARY CORRECTIVE ACTION(S): (1) Immediate project-wide distribution of additional quality assurance documents submitted by participating laboratories (Appendices K, L, and M) and Sections 2, 3, 17 and 18 (Change Form #1) of the CIBA-GEIGY QA Documents Supplement #1 is needed. (2) Additional informational corrections are needed in the CIBA-GEIGY QA Documents Supplement #1, Sections 4 (pages 4-2 and 4-11) and Section 9 (page 9-1). The implementation of the changes in Sections 4 and 9 will be made when the first revisions are made to these sections based on a technical change. Note that technical changes to these sections are expected within the next 90 days.

Iliana Isldi	5/26/92
Project Manager Responsibility: National Service Contract Administrator	Date /
CIBA-GEIGY Project Coordinator	5/29/92 Date
APPROVED (if technical change):	
USEPA Project Manager	Date

CRANSTON QAPJP TRACKING FORM

No. 2 Date: 05/26/92

PERSON REQUESTING REVISION: Thomas Barber

RESPONSIBILITY OF REQUESTOR IN CRANSTON PROJECT:
Group Leader, Analytical Chemistry, CIBA-GEIGY Corporation

NATURE OF CHANGE TO QAPIP:

Type: 1. Major___ Minor_X (check one)

2. Informational Technical X (check one)

Section(s): 2 and Appendices C and D

of Document: CIBA-GEIGY QA Documents Supplement # 1 - APPENDIX H

REVISIONS: (1) Addition of two Standard Operating Procedures (SOPs) for the quantitative determination of silver and zinc by Zeeman Graphite Atomic Absorption using low concentration digestion techniques. (2) Addition of Alan Lynch, Inorganics Laboratory Supervisor, to CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan distribution list.

<u>REASON FOR CHANGE</u>: (1) Low detection limits are required for Pawtuxet River sampling program. (2) Additional personnel are involved in the project.

<u>EFFECTS OF CHANGE</u>: Addenda to CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan (Appendices C and D)

NECESSARY CORRECTIVE ACTION(S): Project-wide distribution of Section 18 (Change Form #2) with following attachments pertaining to Appendix H of CIBA-GEIGY Quality Assurance Documents - Supplement #1:

- (1) Section 2 of CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan
- (2) Appendices C and D of CIBA-GEIGY Analytical Chemistry Quality Assurance Project Plan

Shana Baldi	5/26/92
Project Manager Responsibility: National Service Contract Administrator	Date
CIBA-GEIGY Project Coordinator	5/29/92
CIBA-GEIGY Project Coordinator	Date '
APPROVED (if technical change):	•
USEPA Project Manager	Date

REVISED APPENDIX H

TABLE OF CONTENTS

SECTION		REVISION	<u>PAGE</u>
1.0	Title Page	0	1-1
2.0	Table of Contents	1	2-1
3.0	Project Description	0	3-1
4.0	Project Organization and		_
	Responsibilities	0	4-1
5.0	Quality Assurance Objectives	0	5-1
6.0	Sampling Procedures	0	6-1
	Sample Custody	0	7-1
8.0	Calibration Procedures and		
	Frequency (Lab and Field)	0	8-1
9.0	. ,	0	9-1
10.0	Data Reduction, Validation,		
	and Reporting	0	10-1
	Internal QC Checks	0	11-1
12.0	Performance and Systems Audits	0	12-1
	Preventative Maintenance	0	13-1
14.0	Specific SOPs Used to Assess Data		
	Precision, Accuracy, Representativenes		
	and Completeness	0	14-1
15.0	Corrective Action for Out-of-Control		
	Situations	0	15-1
	QA Reporting Procedures	0	16-1
17.0	List of Current Personnel for Key		
10.6	Positions	0	17-1
18.0	QAPjP Changes	0	18-1

Appendices:

- A. Required Containers, Preservation Techniques, Minimum Sampling Volume and Holding Times
- B. PCB in Soil Screening Procedure, EPA Region I
- C. Quantitative Determination of Silver by Zeeman Graphite Atomic Absorption Using Low Concentration Digestion Techniques, CIBA-GEIGY Analytical Procedure G1000
- D. Quantitative Determination of Zinc by Zeeman Graphite Atomic Absorption Using Low Concentration Digestion Techniques, CIBA-GEIGY Analytical Procedure G1001

AC QA PROJECT PLAN SECTION NO. 2 REVISION NO. 1 05/26/92 PAGE 2 OF 2

REPORT DISTRIBUTION:

Mark Houlday (for Project-wide distribution)
Woodward-Clyde Consultants
201 Willowbrook Boulevard
Post Office Box 290
Wayne, NJ 07470
Voice: (201) 785-0700
FAX: (201) 785-0023

Thomas Barber, Group Leader (919) 632-7297
Alan Lynch, Inorganics Laboratory Supervisor (919) 632-7072
Daniel Winn, Organics Laboratory Supervisor (919) 632-7312
Diana Baldi, NSCA (919) 632-7506
Ciba-Geigy Corporation
410 Swing Road
Greensboro, NC 27409
FAX: (919) 632-2048

Frank Saksa Quality Assurance Officer Ciba-Geigy Corporation Route 37 West Toms River, NJ 08753 Voice: (908) 349-5200 X2789

FAX: (908) 505-9195

CIBA-GEIGY CORPORATION CORPORATE ENVIRONMENTAL TECHNOLOGY CENTER ANALYTICAL CHEMISTRY GROUP

QUALITY ASSURANCE PROJECT PLAN APPENDIX C

Corporate Environmental Technology Analytical Chemistry Greensboro, NC

STANDARD OPERATING PROCEDURE

Procedure No. : G1000

Date: 5/5/92

Title:

THE QUANTITATIVE DETERMINATION OF SILVER IN GROUNDWATER AND WASTEWATER BY ZEEMAN-GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY USING LOW CONCENTRATION DIGESTION TECHNIQUES.

Author

Alan Lyngh

Approved By:

Group Leader

QA/QC Officer

PURPOSE:

This method describes the procedure for the sample preparation and quantitative determination of silver at low concentrations using a Perkin-Elmer 5100 Zeeman-graphite furnace atomic absorption spectrometer with an AS-60 autosampler.

SCOPE:

This method is applicable to the analyses of all groundwater and wastewater samples.

SAFETY:

When handling concentrated mineral acids and hydrogen peroxide, a face shield, rubber gloves, and a lab coat/apron are to be worn. In addition, all laboratory work is to be conducted in a fume hood.

SAMPLING AND STORAGE:

Each sample is to be preserved with nitric acid (Optima grade) to a pH<2, and stored in a glass jar with a Teflon lined cap or an acid-cleaned plastic container for a maximum holding time of 6 months. A minimum of 250 ml of sample is needed for this analysis.

REAGENTS:

- Concentrated Nitric Acid, Optima grade, Fisher Scientific A467, or equivalent. Discard after 1 year.
- 2. Silver standard reference solution, 1000 ppm certified. The standard solution should be discarded after the expiration date indicated on the bottle, or after one year has passed, whichever comes first. Prepare a 10 ppm solution by dilution of 5 ml of 1000 ppm stock into 2% nitric acid (Optima grade) in a 500 ml

volumetric flask. Prepare every 30 days.

- 3. Intermediate silver stock solution I (500 ppb). Prepare by dilution of 5.0 ml of 10 ppm stock solution into a 100 ml volumetric flask and bring to volume with 2% nitric acid (Optima grade). Prepare daily.
- 4. ASTM Type I water.
- 5. Hydrogen Peroxide 30%, Ultrex grade, J.T. Baker Chemical JT5170, or equivalent.

APPARATUS:

- 1. Perkin-Elmer Model 5100 Zeeman-Graphite spectrophotometer with AS-60 autosampler or equivalent.
- 2. Acid-cleaned labware (Teflon beakers, Teflon autosampler vials, Class A volumetric flasks, pipets, etc.)

CLEANING OF GLASSWARE:

All labware used for low concentration analyses should be separated and stored away from labware used for routine analyses. All labware should be prepared by the following sequence:

- 1. Wash all labware in hot soapy water. Teflon containers should be gently washed with a soft sponge.
- 2. Rinse thoroughly with distilled water.
- 3. Store all labware in 10% nitric acid.
- 4. Before use, rinse thoroughly with ASTM Type I water.

SAMPLE PREPARATION:

All acid preserved samples being analyzed for silver content should be digested by the following procedure:

- A 100 ml representative aliquot is transferred to a 400 ml Teflon beaker. Add 2 ml of concentrated nitric acid (Optima grade). Add 2 ml of 30% hydrogen peroxide (Ultrex grade)
- 2. Cover with a Teflon watch glass. Heat the solution for 2 hours at approximately 95° C, or until the total volume has been reduced to approximately 40 ml. Do not boil the solution.
- Cool the solution to room temperature.
- 4. Quantitatively transfer the digestate to a Class A 100 ml

volumetric flask and bring to volume with Type I water. Any solids in solution after digestion can be allowed to settle overnight, or centrifuge the solution in an acid-cleaned tube. If centrifugation is used to remove solids, then the preparation blank must be carried through the same procedure.

5. For each group of samples processed or every ten samples (whichever is less), preparation blanks, spiked samples, and replicates are to be carried though the digestion procedure.

QUALITY CONTROL:

- 1. Calibration standards consisting of a reagent blank, 5.0, 10.0, 20.0, and 25.0 ppb silver are prepared for each run. Prepare by dilution of 1.0, 2.0, 4.0, and 5.0 ml of intermediate stock solution I into separate 100 ml volumetric flasks, and bring to volume with a mixture of 2% HNO3 and 2% H2O2. To verify the calibration standards, run a quality control sample from a second source (source other than used for calibration, such as an EPA known) immediately after each calibration. Control limits are ± 10% of the true value.
- 2. Analyze an instrument check (near the midpoint of the calibration curve) at a 10% frequency. Control limits are \pm 10% of the true value.
- 3. A sample replicate analysis (duplicate sample) is to be performed with each group or batch of 10 samples. A control limit of 20 % RPD (relative percent difference) will be observed in cases where the sample result is at least 5 times the Method Detection Limit.
- 4. A matrix spiked sample is to be run with each group or batch of 10 samples. Spiking levels should be 0.5 to 1.0 times the expected sample level. If unsure of the expected sample level, spike with 10 ppb. If the percent recovery does not fall within 75 to 125 % then a post spike analysis (≈ 1.0 X sample result) should be performed. If the post spike analysis is out of control, then Method of Standard Additions (MSA) should be used to determine sample concentration.
- 5. All furnace analyses require duplicate sample injections (except for Method of Standard Additions). A control limit of 20 % RSD will be observed in cases where the sample average is at least 5 times the Method Detection Limit.
- 6. Method detection limits (MDL) are determined by analyzing seven replicates of a known standard that is 3 to 5 times the anticipated (or previously determined) detection limit. The detection limit is calculated to be 3 times the standard deviation

of the seven replicate analyses.

- 7. The MDL for this technique is 0.20 ppb
- 8. All QC data should be maintained and available for easy reference or inspections.

SAMPLE ANALYSIS:

For specific details concerning the operation of the Zeeman graphite 5100 AAS and autosampler, refer to the instrument operation manuals.

CALCULATIONS:

- 1. No calculations for concentrations are necessary.
- 2. Determination of relative percent difference (RPD) for replicates (duplicate samples):

- 3. Percent Recovery determination:
 - % Recovery = Spiked Sample Result Sample Result X 100
 Spike Added
- 4. Percent Relative Standard Deviation (% RSD):
 - % RSD = <u>Sample Standard Deviation</u> X 100 Average Sample Concentration

TRAINING

All personnel performing this analysis must undergo a training procedure conducted by an experienced technician and observed to insure proper methodologies are adhered to. Also, once training is completed, the addition of the analysis must be documented in the individual's personnel file.

REFERENCES

USEPA Methods for Chemical Analyses of Waste and Wastes, EPA 220.2, EPA 600/4- 79-020.

USEPA Methods for Evaluating Solid Waste, EPA SW 846, Dec 87

USEPA Contract Laboratory Program SOW, Document Number ILM02.0

CIBA-GEIGY CORPORATION CORPORATE ENVIRONMENTAL TECHNOLOGY CENTER ANALYTICAL CHEMISTRY GROUP

QUALITY ASSURANCE PROJECT PLAN APPENDIX D

Corporate Environmental Technology Analytical Chemistry Greensboro, NC

STANDARD OPERATING PROCEDURE

Procedure No.: G1001

Date: 5/5/92

Title: THE QUANTITATIVE DETERMINATION OF ZINC IN GROUNDWATER AND WASTEWATER

BY ZEEMAN-GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROSCOPY USING LOW

CONCENTRATION DIGESTION TECHNIQUES.

Author

Alan Lyngh

Approved By:

Group Leader

QA/QC Officer

PURPOSE:

This method describes the procedure for the sample preparation and quantitative determination of zinc at low concentrations using a Perkin-Elmer 5100 Zeeman-graphite furnace atomic absorption

spectrometer with an AS-60 autosampler.

SCOPE:

This method is applicable to the analyses of all groundwater and wastewater samples.

SAFETY:

When handling concentrated mineral acids and hydrogen peroxide, a face shield, rubber gloves, and a lab coat/apron are to be worn. In addition, all laboratory work is to be conducted in a fume hood.

SAMPLING AND STORAGE:

Each sample is to be preserved with nitric acid (Optima grade) to a pH<2, and stored in a glass jar with a Teflon lined cap or an acid-cleaned plastic container for a maximum holding time of 6 months. A minimum of 250 ml of sample is needed for this analysis.

REAGENTS:

- 1. Concentrated Nitric Acid, Optima grade, Fisher Scientific A467, or equivalent. Discard after 1 year.
- Zinc standard reference solution, 1000 ppm certified. The standard solution should be discarded after the expiration date indicated on the bottle, or after one year has passed, whichever comes first. Prepare a 10 ppm solution by dilution of 5 ml of 1000 ppm stock into 2% nitric acid (Optima grade) in a 500 ml

volumetric flask. Prepare every 30 days.

- 3. Intermediate zinc stock solution I (500 ppb). Prepare by dilution of 5.0 ml of 10 ppm stock solution into a 100 ml volumetric flask and bring to volume with 2% nitric acid (Optima grade). Prepare daily.
- 4. ASTM Type I water.
- 5. Hydrogen Peroxide 30%, Ultrex grade, J.T. Baker Chemical JT5170, or equivalent.

APPARATUS:

- 1. Perkin-Elmer Model 5100 Zeeman-Graphite spectrophotometer with AS-60 autosampler or equivalent.
- 2. Acid-cleaned labware (Teflon beakers, Teflon autosampler vials, Class A volumetric flasks, pipets, etc.)

CLEANING OF LABWARE:

All labware used for low concentration analyses should be separated and stored away from labware used for routine analyses. All labware should be prepared by the following sequence:

- 1. Wash all labware in hot soapy water. Teflon containers should be gently washed with a soft sponge.
- 2. Rinse thoroughly with distilled water.
- 3. Store all labware in 10% nitric acid.
- Before use, rinse thoroughly with ASTM Type I water.

SAMPLE PREPARATION:

When preparing samples for zinc analysis by graphite furnace, extreme care must be taken to minimize sample contamination. Work in a freshly cleaned fume hood that is isolated from other routine digestions taking place at the same time. Powder-free clean room gloves should be worm at all times. All acid preserved samples being analyzed for zinc content should be digested by the following procedure:

- 1. A 100 ml representative aliquot is transferred to a 400 ml Teflon beaker. Add 2 ml of concentrated nitric acid (Optima grade). Add 2 ml of 30% hydrogen peroxide (Ultrex grade)
- 2. Cover with a Teflon watch glass. Heat the solution for 2 hours at approximately 95° C, or until the total volume has been reduced to

approximately 40 ml. Do not boil the solution.

- 3. Cool the solution to room temperature.
- 4. Quantitatively transfer the digestate to a Class A 100 ml volumetric flask and bring to volume with Type I water. Any solids in solution after digestion can be allowed to settle overnight, or centrifuge the solution in an acid-cleaned tube. If centrifugation is used to remove solids, then the preparation blank must be carried through the same procedure.
- 5. For each group of samples processed or every ten samples (whichever is less), preparation blanks, spiked samples, and replicates are to be carried though the digestion procedure.

QUALITY CONTROL:

- 1. Calibration standards consisting of a reagent blank, 5.0, 10.0, 15.0, and 20.0 ppb zinc are prepared for each run. Prepare by dilution of 1.0, 2.0, 3.0, and 4.0 ml of intermediate stock solution I into separate 100 ml volumetric flasks, and bring to volume with a mixture of 2% HNO3 and 2% H2O2. To verify the calibration standards, run a quality control sample from a second source (source other than used for calibration, such as an EPA known) immediately after each calibration. Control limits are ± 10% of the true value.
- 2. Analyze an instrument check (near the midpoint of the calibration curve) at a 10% frequency. Control limits are \pm 10% of the true value.
- 3. A sample replicate analysis (duplicate sample) is to be performed with each group or batch of 10 samples. A control limit of 20 % RPD (relative percent difference) will be observed in cases where the sample result is at least 5 times the Method Detection Limit.
- 4. A matrix spiked sample is to be run with each group or batch of 10 samples. Spiking levels should be 0.5 to 1.0 times the expected sample level. If unsure of the expected sample level, spike with 10 ppb. If the percent recovery does not fall within 75 to 125 % then a post spike analysis (≈ 1.0 X sample result) should be performed. If the post spike analysis is out of control, then Method of Standard Additions (MSA) should be used to determine sample concentration.
- 5. All furnace analyses require duplicate sample injections (except for Method of Standard Additions). A control limit of 20 % RSD will be observed in cases where the sample average is at least 5 times the Method Detection Limit.

- 6. Method detection limits (MDL) are determined by analyzing seven replicates of a known standard that is 3 to 5 times the anticipated (or previously determined) detection limit. The detection limit is calculated to be 3 times the standard deviation of the seven replicate analyses.
- 7. The MDL for this technique is 0.20 ppb
- 8. All QC data should be maintained and available for easy reference or inspections.

SAMPLE ANALYSIS:

For specific details concerning the operation of the Zeeman graphite 5100 AAS and autosampler, refer to the instrument operation manuals. Clean the graphite furnace contact rings and install a new graphite tube before each analysis run. Extreme care should be taken when setting up the analysis run to minimize zinc contamination. Powder-free clean room gloves should be worn at all times.

CALCULATIONS:

- 1. No calculations for concentrations are necessary.
- 2. Determination of relative percent difference (RPD) for replicates (duplicate samples):

- 3. Percent Recovery determination:
- 4. Percent Relative Standard Deviation (% RSD):
 - % RSD = <u>Sample Standard Deviation</u> X 100 Average Sample Concentration

TRAINING

All personnel performing this analysis must undergo a training procedure conducted by an experienced technician and observed to insure proper methodologies are adhered to. Also, once training is completed, the addition of the analysis must be documented in the individual's personnel file.

REFERENCES

USEPA Methods for Chemical Analyses of Waste and Wastes, EPA 220.2, EPA 600/4-79-020.

USEPA Methods for Evaluating Solid Waste, EPA SW 846, Dec 87

USEPA Contract Laboratory Program SOW, Document Number ILM02.0

APPENDIX K

Savannah Analytical Services Suite "I" 42 West Montgomery Crossroads Savannah, GA. 31406

May 1, 1992

Kathy Gallant Ciba-Geigy Corporation P. O. Box 18300 410 Swing Road Greensboro, N. C. 27419

Dear Kathy:

Enclosed are the responses to the statements listed in the Sample Analysis Section of Data Collection Quality Assurance Plan from pages 39-40 of the Cranston Administrative Order.

If there are any other questions, or if there is anything else needed from SAS, please call. Thank you.

Sincerely,

Steve Bishop --

SB:ms

Enclosures

d. Sample Analysis

i.

aa. Steve Bishop

bb. SAS Standard CHN Report Forms

cc. NA

ii. Included in SOP. Filtered samples low temperature dried and stored in a dessicator until analysis.

iii. Included in SOP

iv.

aa. Included in SOP

bb. Included in SOP

cc. NA

dd. Carbon - 0.1 ug/l Nitrogen - 0.1 ug/l Hydrogen - 0.1 ug/l

v. Included in SOP

vi. Submitted after sample analysis

vii. NA

viii.	Interval	Procedure .
	200 cycles	Replace Reduction Tube
	150 cycles	Replace Combustion Tube

Helium Pressure below 300 psi

Replace He Cylinder

Oxygen Pressure below 50 psi

Replace O Cylinder

As indicated by

chemical color change

Replace CO2 & H20 Traps

As indicated by

chemical color change

Replace He & O Scrubbers

Every 3 months

Lubricate Fan Motor

Every 6 Months of if indicated by leaks

Check solenoid valves, clean and replace if necessary.

Same frequency for system O-rings.

 Savannah Analytical Services
 42 West Montgomery Crossroad, Suite I Savannah, GA 31406

S O P - CHN Analysis (Aqueous Samples)

Sample Prep

- 1. Before analysis of any sample, cut pieces of Aluminum Foil into approximately 1" squares. These will be used to enclose samples for particulate analyses.
- Pre-combust glass fiber filters (25mm) in a muffle furnace (400-450 degrees C) for 4-6 hours. Remove and store in a dessicator until needed.
- 3. Filter measured sample onto 25mm glass fiber filters using a vacuum pump with side arm flask or manifold system (pump pressure below 10 psi). The volume to be filtered is determined by expected concentration of particulate in the sample. For example:

Partic	<u>ulate Concentration</u>	Volume To Filter	-
	1-5 mg	500 ml - 1 liter	-
-	>5 <10 mg	250 ml - 500 ml	
•	>10 ma	25 ml - 100 ml	

the side containing the particulate to the inside. This keeps the sample from being scraped off in the storeage container. Wrap lightly in one of the aluminum foil squares. [If the filters are to be dried immediately, they don't have to be folded and wrapped. Just lay them in the oven on a square of al foil in a labeled container.]

- 5. Lable container with Sample No. and volume filtered.
 Using forceps transfer foil package containing sample to the labelled container.
- 6. Store in a freezer (< 4 degrees C) until ready to dry or dry immediately in a low temperature oven (50-60 deg.C) for 2-3 hours. If drying frozen filters, let samples thaw slightly at room temperature, unfold and proceed with drying as described above in this paragraph.
- 7. After drying, use forceps to wrap samples tightly in al foil squares so that they will fit into a CHN Sample Ladle (technique similar to rolling an old fashioned cigarette!)
- Place sample in a dry, labelled container and store in a dessicator until ready to analyze.

CHN Procedure

- 1. Adjust He regulator to 18 psi and Oxygen regulator to 30 psi.
- 2. Rotate indicator wheel on 240 Elemental Analyzer to 2 (Hepurge) for 5 minutes.
- 3. Rotate indicator wheel through 0 to 1 (Oxygen purge) for 3 minutes.
- 4. Rotate indicator wheel to 0.
- 5. Place a small amount of Acetanilide (1-3 mg) on an al foil square and roll up. This is an unweighed standard used to "condition" the instrument.
- 6. Place the unweighed standard into a sample ladle, remove the entrance plug from the 240 analyzer, and insert the ladle into the combustion tube.
- 7. Push the START button Sample will now go through the analysis cycle (takes approximately 12 minutes).
- 8. At 5 minutes into the cycle the "ZERO" values for N, C, and H will be displayed and printed. At 10 minutes into the cycle the "READ" values will be displayed and printed (these values are in units of micro volts).
- 9. When the indicator wheel rotates to 0, the ladle can be removed and a new sample can be inserted into the combustion tube.

- 10. The second run of the day is a foil blank (just a square of the pre-combusted al foil rolled so that it will fit into the ladle.
- 11. Again as the cycle proceeds, "ZERO" and "READ" values will be displayed and printed. For the Foil Blanks, the "ZERO" value for N is subtracted from the "READ" value for N, with the same procedure used for C and H.

READ VALUE - ZERO VALUE = BLANK VALUE

- 12. Rotating Unweighed Standards and Foil Blanks continues until consistent blank values for N (<5%), C(<5%), and H (<10%) are reached.
- 13. Then a weighed standard of Acetanilide is run through the instrument. The "READ" value of the weighed standard minus the "ZERO" value minus the "BLANK" value gives the Total Signal for the standard (units of micro volts).

READ VALUE - ZERO VALUE - BLANK VALUE = TOTAL SIGNAL

- 14. The TOTAL SIGNAL is divided by the theoretical weight in micrograms of N(10.36%), C(71.09%), and H(6.54%) present in Acetanilide. This number is known as the "SENSITIVITY" and is denoted as the "K" factor with units of uV/ug.
- 15. The K Factors must be within the following ranges:

K N = 6 to 10 uV/ug

K C = 15 to 25 uV/ug

K H = 44 to 76 uV/ug

- 16. When the weighed standard meets these criteria the instrument is calibrated and ready for sample runs. A sample of the Calibration Data Sheet is included.
- 17. Samples can now be analyzed. The procedure is the same as for the WEIGHED STANDARDS listed in step 13 with one exception. The TOTAL SIGNAL of the samples is divided by the K FACTOR as follows:

SAMPLE TOTAL SIGNAL/K FACTOR = SAMPLE CONCENTRATION
Units for the calculation are as follows:

uV/(uV/ug) = ug CONCENTRATRION IN SAMPLE

A sample of the data sheet for samples is also included.

18. The concentration in micro grams is then divided by the volume filtered. Units for sample concentration are now in ug/liter.

ug CONC./VOLUME FILTERED = UG/LITER





Ms. Kathy Gallant Ciba-Geigy Corporation P.O. Box 18300 410 Swing Road Greensboro, NC 27419

Dear Ms. Gallant:

In response to your letter of April 28, 1992 regarding information for your data collection quality assurance plan, as identified in the Ciba-Geigy, Cranston, RI, Facility consent order.

c. Field measurements

ix. Decontamination of sampling equipment: sampling containers are washed with laboratory soap and tap water. Rinsed 3 times with tap water and 5 times with DI water.

d. Sample Analysis:

- i. Chain of Custody Procedures
 - aa) The lab manager or sample custodian is authorized to sign for incoming samples, sample condition and identification are noted by the sample custodian.
 - bb) All sample information is entered into the laboratory information management system. Hard copies of laboratory inventory sheets are kept on file in the laboratory managers office. Sample bottles are serially numbered based on the laboratory inventory sheets.
 - cc) All samples are retained in a secure area at 4°C where applicable. Secure area has restricted access in order to maintain sample integrity. Samples are dispersed for analysis by the laboratory manager or the appropriate laboratory supervisor.

ii. Sample storage

Addressed in cc) above

iii. Sample preparation

As per EPA method 160.2 non-filterable residue, gravimetric, dried at 103 - 105°C

iv. Analytical Procedure

- aa) As per EPA method 160.2 above.
- bb) River water
- cc) No interferences expected
- dd) Precision, low level = 25%, high level I 10%*
 Accuracy data on actual samples cannot be obtained.
- ee) Method detection limit. 0.5 mg/l

v. Calibration procedures

Analytical balances are calibrated daily using class S weights near the weighing range expected. Monthly, the balance is checked using class S weights from 1 mg to 100 mg. Annually, the analytical balances are serviced by a qualified service representative and checked with a set of NBS weights. Oven temperatures are checked daily.

vi. Data Reduction

Data reduction follows the method referenced previously. (EPA 160.2)

Validation - all analyses performed are reported on standardized reporting sheets designed for the analyte of concern. Each time a test is performed, the analyst enters data concerning the EPA method used, all calculations and raw data readings, and explanation of any preparation involved, the final result and the analyst's signature and date.

Final reported concentrations are calculated and reviewed based upon the information presented on the work sheet.

*High level is defined as greater than 20 times the M.D.L.

vii. Performance and System Audits

The laboratory actively participates in external performance evaluation programs for water pollution, drinking water quality, RCRA, and NPDES permitting. These programs, identified as the WP (Water Pollution) and WS (Water Supply) series are administered by the EPA. Performance evaluation samples for each series are analyzed every six months. The results of these analyses are sent to the EPA for evaluation. The EPA evaluations are then sent directly to the Rhode Island Department of Health which in turn forwards the information to the laboratory. Any problems or errors are investigated by the QA Officer and the Laboratory Manager and corrective action is taken.

All new instrument systems are thoroughly checked with known standards and samples and the data quality evaluated before the system is used to evaluate unknown samples. This information combined with internal data described in the previous sections make up the data which is used by the quality assurance officer and the laboratory director in determining systems QC.

viii. Not applicable

A copy of our standard operating procedure for total suspended solids is attached. If you have any questions, please feel free to call either Mike Rose or myself.

Sincerely,

Jerry Zschau

Marketing Manager

JZ/djr

TOTAL SUSPENDED SOLIDS (Dried at 103-105 Degrees C)

REFERENCE: Methods for Chemical Analysis of Water and Wastes, EPA 600/4-79-020, Revised March, 1983, Method 160.2.

METHOD SUMMARY:

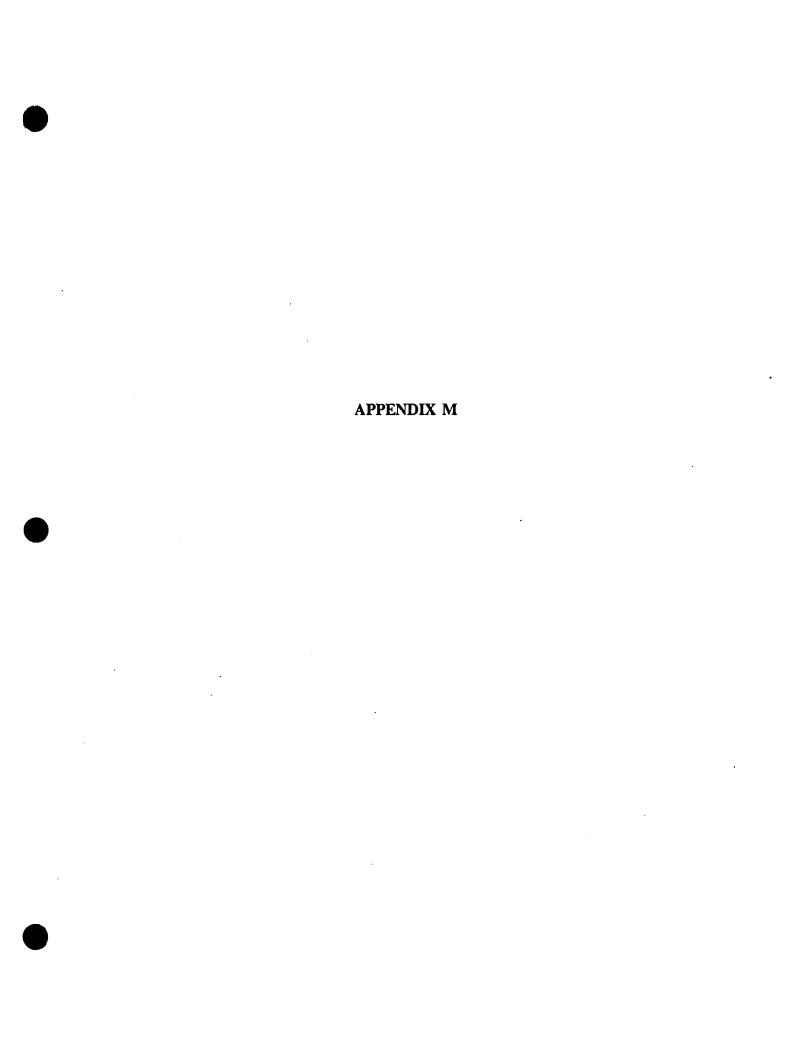
- Prepare 47mm glass fiber filters for use by first washing them with deionized water in the fitration apparatus to be used. Place the disk in the membrane filter funnel with the wrinkled side up into the filtration apparatus and wash with about 50 mls of water.
- 2. Place the washed filters into small aluminum weighing dishes and dry for at least several hours at 103-105 degrees C to assure complete dryness. Remove the filters and store in a desiccator until ready to use. Weigh just before using with an analytical balance to five places.
- 3. Assemble membrane filter funnel with the filter in place and connect to the suction flask and vacuum. Filter a measured volume of a well mixed sample through the glass fiber filter and follow with three successive washings of 30 ml portions of DI water, washing the graduated cylinder and sides of the membrane filter funnel. (Choose a sample volume that is large enough to be a representative sample and small enough to limit the residue to about 200 mg.)
- 4. After the washings, continue the suction until the filtration is complete and let it continue for 3 minutes to assure dryness. Remove the filter and place in a small aluminum weighing pan and dry for at least 1 hour in the oven at 103-105 degrees C.
- 5. Remove the sample from the oven and cool to room temperature before weighing. Repeat the drying cycle until a constant weight is obtained or the weight loss is less than 4% the previous weight.

CALCULATION: TSS, mg/L = $(A - B) \times 1000 \times 1000$ sample vol, ml

A= wgt. of filter + dried residue, g. B= wgt. of filter, g.

INTERFERENCES: Large floating particles, excessive residue, surfactants, high dissolved solids.

PRESERVATION: Nonpreserved, 4C. HOLD TIME: 7 Days
DETECTION LIMIT: <0.5 mg/L (dependent upon sample volume)</pre>



QUALITY ASSURANCE PLAN

FOR

CIBA-GEIGY CORPORATION

LOW LEVEL PCB ANALYSIS FOR WATER

April 29, 1992

NORTHEAST ANALYTICAL, INC 301 NOTT STREET SCHENECTADY, NY 12305 518-346-4592 FAX: 518-381-6055

INTRODUCTION

INTRODUCTION

This document plan sets forth the methodology and necessary procedures and documentation to generate analytical data for trace Polychlorinated Biphenyl (PCB) measurement in environmental samples. To insure accountability and sample integrity the following quality assurance objectives will be implemented. This includes quality control documentation, sample custody records, sample tracking records, data validation, and data reporting.

SAMPLE RECEIPT AND CHAIN OF CUSTODY PROCEDURES

SAMPLE RECEIPT AND CHAIN OF CUSTODY PROCEDURES

Northeast Analytical's sample custodian is Ms. Tracy M. Bengtson. Tracy has held this position for 2 years and is well versed in sample receipt, chain of custody procedures, in-house ID assignment, log book maintenance, sample handling, storage, and distribution. Examples of the following are provided: sample receipt, login, lab tracking forms, and chain of custody forms.

After samples are assigned a unique sample ID, they are stored in either a walk-in cooler or appropriate sample refrigerator (i.e. volatiles). The lab manager is notified that samples have arrived and analyte(s) requested. Sample ID's, along with other pertinent information, are entered into our computerized sample tracking system. A printout of the sample tracking computer file is included for review.

Individual folders are generated to track each group of samples for analysis which include a copy of the original COC, any additional letters from the client, in-house control sheets, and a sample analysis sheet. The lab manager reviews each folder and assigns sample folders to the appropriate analytical chemist trained in preparation; extraction; and analysis for the analytes requested. The analytical chemist obtains the group of samples listed in the work folder from storage and proceeds with the sample analysis. The chemist initiates a sample lab sheet to record all

pertinent information during sample preparation, including weight or volume, date of extraction, original volume of extract, and dilutions prior to analysis, surrogate added before extraction, and internal standard amounts used for analysis. A copy of the sample lab form is provided for review.

All samples are extracted within EPA prescribed holding times for the particular analyte. All samples are analyzed within EPA prescribed holding times for the particular analyte. If there is any deviation from meeting prescribed hold times for any reason, that client is informed immediately for instructions as how to proceed.

SAMPLE RECEIPT

SAMPLE RECEIVING

ALWAYS WEAR SAFETY GLASSES IN THE LAB, AND WHEN HANDLING SAMPLES ALWAYS WEAR DISPOSABLE GLOVES.

I. CLIENT RELATIONS:

A. When client arrives with samples, welcome them and help them unload the samples. Ask the client if they brought a chain of custody record with them, if not fill out one.

II. CHAIN OF CUSTODY:

- A. Review chain of custody with client, being sure that there are no discrepencies in the sample Id numbers or the number of samples, be sure to note parameters.
- B. Check sample for leakage, cracks, or broken caps.
 Be sure that sample contains preservative if necessary as the preservative effects the holding time
 ie.: HCl for Volatiles {see attached sampling guide}
- C. Record any of this information on the chain of custody
- D. You and the customer should Sign, date and time the custody.
- E. Give the pink copy of the chain of custody to the client.
- F. Thank the client for bringing the sample, and tell them
 when they can expect the results.
 {Normal turn-around-time = two weeks}
 {Rush turn-arounds should be approved with Lab manager}

SAMPLE LOG-IN

SAMPLE LOG IN

ALWAYS WEAR SAFETY GLASSES IN THE LAB, AND WHEN HANDLING SAMPLES ALWAYS WEAR DISPOSABLE GLOVES.

I. ASSIGNING NEA ID NUMBERS:

- A. Refer to SAMPLE TRACKING LOG BOOK AND CHAIN OF CUSTODY to aquire your next available NEA SAMPLE ID NO.
- B. Assign a NEA sample Id# for each sample brought by client directly next to the customer ID# on the Chain of Custody. *Check to be sure the numbers are accurate and in order.
- C. Put samples in the order they appear on the chain of custody.
- D. Assign each sample our ID#, checking your work as you do so. Write our sample ID# on the label, cap and jar if possible.
- E. After logging in all the sample you should again check to be sure that all samples are logged in, that all the numbers are legible and accurate.
- F. Place samples in a box, label, and place in storage.
- F. Place original chain of custody in the chain of custody log book, most recent custody should be placed in front of book.

II. PROGRESS TRACKING FOLDERS:

- A. One copy of the chain of custody, progress tracking sheets, {must be filled in: see attached example} and any other information should be put in a folder {label w/client:NEA ID#s] and placed on the folder rack.
- B. Always make Laboratory manager aware of incoming samples.
- C. Write Client, Parameter, and NEA sample ID#s on the wall calender in the hallway, on the date it is due.
- D. Samples must then be logged into our Sample Tracking Log Book by computer: C:\SC4\LAB1991F.CAL {file varies with year}.

 *IF UNABLE TO DO THE COMPUTER LOG, HAND WRITE NEA SAMPLE ID

 NUMBERS IN LOG BOOK SO THEY ARE RECORDED AS TAKEN,
 AND WILL NOT BE USED AS THE NEXT AVAILABLE NUMBERS.

PROGRESS TRACKING FORMS

CLIENT		
NEA ID		
•		
DATE RECEIVED:		
REQUESTED COMPLETION DATE:	(CIRCLE) NORMAL \	RUSH
PARAMETERS TO BE PER	FORMED	
PARAMETER ** CDA METHODOLOGY		
PARAMETER *EPA METHODOLOGY 1		
2		
3		
4		
5		
7		
8	· · · · · · · · · · · · · · · · · · ·	<u></u>
9		
1.0		
TOTAL NUMBER OF ANALYSES:	•	
*If applicable		
Completion Date of all Analyses:	, , , , , , , , , , , , , , , , , , , 	·
Reviewed by:		·

INTERNAL PROJECT TRACKING SHEET

NEA SAMPLE ID:	
CLIENT:	
SAMPLE MATRIX:	
ANALYSES REQUESTED:	
	· · · · · · · · · · · · · · · · · · ·
	•
QA:	•
DATE SAMPLES DRIED:	ANALYST:
EXTRACTION DATE:	ANALYST:
,	
CLEAN-UP PROCEDURE:	
	·····
CLEAN-UP COMPLETION DATE:	ANALYST:
SCREEN DILUTION:	ANALYST:
DATE PROJECT SENT FOR GC SCREEN:	ANALYST:
DDO IECT COMPLETION DAME.	
PROJECT COMPLETION DATE:	ANALYST:
COMMENTS/NOTES:	
·	

CHAIN OF CUSTODY FORM

		LAB SERVICI Y 12305 (518) 346-4		Ref	frigerator #								<i></i>	NEA Use Only
				#/Т	Type Container			 		<u> </u>	+-	+		Samples were:
CLIENT Work				voi	lume	+		 	 		+-	+-+		
Order Date		Date		Pre	servative	+		-			+-	+-+		1 Shipped or Hand-Delivered Notes:
Received Due RFW Contact Client Contact and Client Phone #			ANALYSES REQUESTED									2 Ambient or Chilled		
NEA Use Only Lab ID	Cli	lient ID/Description		Matrix *	Date Collected					l		+		3 Received Broken/
												+++		Leaking (improperty sealed): Yes No
	1													Notes:
	+				 	+	لــــــــــــــــــــــــــــــــــــــ				\Box			
					 	+-+		-				++		4 Properly Preserved: Yes No
							, — —				 	+-+		Notes:
	 								<u>'</u>		<u></u>	+		- Indicate Helding
,	 	· · · · · · · · · · · · · · · · · · ·			 		·		\Box					 5 Received Within Holding Times: Yes No
	 			, !		-		<i></i>			<u> </u>			Notes:
				, ———J				,				+		COC Tape was:
								,—		-		-		1 Present on Outer
	ļ						·		丁					Package: Yes No 2 Unbroken on Outer
	<u> </u>									\Box	$\overline{}$			Package: Yes No 3 Present on Sample:
MATRIX: S = Soil SE = Sediment SO = Solid	W = Water O = OII A = Air	F = Fish _ = EP/TCLP (= Other	CLP Leachate					<u>. L.</u>	Yes No 4 Unbroken on Sample: Yes No Notes:					
Item/Reason	Relinquished by	Received by	Date	Time	Item/Reason	Reli	linquishe	ed by	Re	ceived b	by T	Date	Time	<u> </u>
			<u> </u>			 			_ 					COC Record was:
	1		ı!	[/		i					7		 	Present upon receipt of samples: Yes No
		T			1	,				. 	+		 '	Discrepancies between Sample Labels and COC
	1		,								-+		 '	Record? Yes No Notes:
	4	-									ſ	•	1	Notes:

•)

COMPUTERIZED LAB TRACKING PRINTOUT

NORTHEAST ANALYTICAL INC. SAMPLE TRACKING LOGBOOK

FILE: S:\COMMON\LABTRACK\LAB1992A.WK3

DATE: 03/13/92 UPDATE: 04/30/92

ATE	TIME	NEA	CLIENT SAMPLE	SAMP	PARAMETER TO	STOR.	DATE
ec'd	Rec'd	ID#	ID NUMBER		BE PERFORMED	AREA	DATE
	, , , , , , , , , , , , , , , , , , ,	92001451		_WA18		AUEN	COMP.
1		92001452				 	
		92001453		 		 	
		92001454		 			
1		92001455				 	
		92001456				 	
P		92001457		 		 	
		92001458		 			\vdash
		92001459				 	
		92001460			-	 	
		92001461				 	
		92001462				 	
		92001463				 	
		92001464					
		92001465			·		<u>├</u>
		92001466				 	
1		92001467					
		92001468					
		92001469			1		
		92001470				;	
		92001471					
		92001472				:	
		92001473				<u> </u>	
		92001474					
		92001475				1	
		92001476					
'		92001477					
		92001478					
<u></u>		92001479					
l		92001480					
		92001481					
		92001482	<u> </u>				
		92001483	· · · · · · · · · · · · · · · · · · ·				
		92001484					
,		92001485					
		92001486					
'		92001487			•		
		92001488	<u></u>				
 		92001489				<u> </u>	
I		92001490	·····			<u> </u>	
		92001491					
		92001492	· · · · · · · · · · · · · · · · · · ·				
	·	92001493		_		ļ	<u> </u>
		92001494				1	
		92001495	<u>.</u>				
		92001496					
l		92001497					
		92001498					
<u></u>		92001499					
		92001500		L		<u> </u>	

SAMPLE LAB FORM

NORTHEA	STANALYTICAL, INC.	Client:						
CAPILLARY G	SC LAB SHEET							
Set volume ex	straction methods (rev 2, 3/92)							
NEA ID	CUSTOMER ID	EXTRACT DATE	WEIGHT(g) VOL(mL)	INITEXT VOL (mL)	SCRN DIL	SCRN AMT (ppm)	FINAL DILUTION (from initial ext)	FINAL MULTIPLIEF
								
	·							
			· ·					
	· · · · · · · · · · · · · · · · · · ·							
				-				
-								-
		-						
-								
	· · · · · · · · · · · · · · · · · · ·							
				· ·	··.			
nments:								

SAMPLE PREPARATION METHODOLOGY

Standard Operating Procedure : H2OEXT.SOP

Northeast Analytical, Inc. -1- NEA-608CAP REV. 2.0 8/89

Lab Method NEA-608CAP

Congener-Specific Polychlorinated Biphenyl (PCB) Analysis

Method for Congener-Specific Polychlorinated Biphenyl (PCB)
Quantification and Identification by Capillary Column/Gas
Chromatography with Electron Capture Detection

1.0 Scope

- This method is applicable in the determination and quantification 1.1 of Polychlorinated Biphenyls (PCB) in sediments, soils, and water. This method is a congener-specific determination, employing a high resolution fused-silica capillary chromatographic column. The method includes guidelines set forth in the document: "Quality Assurance Plan, Green Bay Mass Balance Study, 1. PCBs and Dieldrin, US EPA Great Lakes National Program Office", prepared by Deborah L. Swackhamer, Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987. document outlines quality assurance and quality control procedures to be followed by laboratories participating in the Green Bay Mass Balance Study. Where applicable, Northeast Analytical, Inc., will incorporate and utilize this information in quality control of data generated. Instrumental analysis and conditions (Mullin, M.D., 1985, PCB Workshop, US EPA Large Lakes Research Station, Grosse Ile, MI, June.) cited in the Green Bay Mass Balance Study document will be refined to be applicable to an in-house data management software package.
- 1.2 This method will be applied specifically to the determination of PCBs by congener, in sediments. The results of these analyses will be used in studies related to biodegradation of PCBs. Highly sensitive techniques will generally not be required, since samples will contain appreciable quantities

2.0 Sample collection and preparation

- 2.1 Grab samples should be collected in 1 liter bostontype bottles that have been pre-cleaned to EPA
 specification. Bottle caps should be teflon lined.
 All sampling equipment must be thoroughly
 decontaminated between sampling stations with glass
 distilled pesticide grade acetone prior to use.
- 2.2 Samples must be stored in a cooler with ice or refrigerated at 4°C from time of collection until delivery to the lab. Every effort should be made to deliver the samples to the lab as soon as possible.
- quickly as possible. Samples should be inspected for breakage or leakage, logged-in, and transferred to a storage cooler at 4°C as soon as possible. Every effort should be made to perform the extraction as soon as possible after sampling (no longer than 5 days after receipt of samples at laboratory).

3.0 Extraction

- 3.1 The extraction procedure for aqueous samples is designed to minimize loss of PCBs during the extraction and clean-up procedure. Every effort must be made by the analyst to keep glassware clean and isolated from possible sources of contamination.
- 3.2 Allow the sample to reach ambient temperature and mark the level of the water on the bottle with a felt tip pen. After extraction the volume of water taken for analysis can be determined by refilling the bottle with tap water to the mark and measuring the volume with a 500 mL graduated cylinder.
- 3.3 Remove the cap from the sample bottle and spike the water sample with 0.5 mL of 0.2 ppm surrogate standard(2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) using a 1.0 mL gas tight syringe. At this time spike any matrix spike sample with the appropriate Aroclor or Aroclor mixture. Typically low level water samples are spiked with 0.2 mL of Aroclor 1242 at a concentration of 1.008 ppm in Acetone. Other matrix spikes can be utilized to better match

the Aroclor contamination of the sampling site if that information is available.

- 3.4 Shake the sample bottle for 30 seconds to thoroughly mix the surrogate standard and matrix spike with the water sample.
- Rinse a 2 liter separatory funnel equipped with a teflon stopcock with approximately 40 mL of methylene chloride. Drain and discard the wash solvent. Rinse the stopper with several disposable pipette volumes of methylene chloride.
- 3.6 Carefully transfer the water sample into the separatory funnel, making sure not to splash or allow sample to run down the outside of the glassware. Once the sample is completely transferred and while still inverting the bottle over the separatory funnel, rinse the neck of the sample bottle with several disposable pipette volumes of methylene chloride. This will remove any water droplets adhering to the glass threads at the bottle neck.

- 3.7 Add 40 mL of methylene chloride to the sample container and shake vigorously for 30 seconds. Transfer this solvent to the separatory funnel by pouring. Once again rinse the bottle neck with methylene chloride to insure complete transfer. Rinse the glass joint with methylene chloride. Pre-wet the glass stopper with methylene chloride and quickly install in place. Wetting the stopper with solvent will prevent the glass joint from "freezing" and makes it easier to remove. Shake the separatory funnel vigorously for two minutes, venting the funnel as necessary to release vapor pressure.
- This motion aids methylene chloride trapped on the surface to sink and more completely phase separate.
- 3.9 If an emulsion forms with the sample and solvent try adding two or three spatula scoops of sodium sulfate to facilitate better phase separation.

- 3.10 Slowly drain the methylene chloride layer (bottom layer) into a pre-rinsed 125 mL boston bottle.
 Rinse the stopcock tip into the boston bottle to insure complete transfer.
- 3.11 Starting at step 3.7 repeat the extraction procedure two more times using 25 mL of methylene chloride. Combine all the extractions into the same 125 mL boston bottle. After extraction is complete place the sample in a freezer to chill.
- 3.12 Prepare a chromatography column (24 mm by 400 mm) to dry the extract. Place a small plug of silanized glass wool into the bottom of the column. Add anhydrous sodium sulfate to a depth of 1.5 inches. Rinse the drying column with 50 mL of methylene chloride and discard rinsate. Place a pre-rinsed 250 mL Turbovap vessel under the column to collect sample extract. Align column tip against wall of turbovap vessel so that solvent drains down the side and avoids sample splashing during elution.
- 3.13 Remove the sample from freezer storage and slowly add a 50/50 mixture of anhydrous magnesium sulfate and sodium sulfate to the extract. Immediately cap

the sample bottle and shake vigorously to facilitate drying agent in absorbing residual water that was transferred during extraction. Add drying agent to sample until it becomes free flowing and no water is visible. Proceed slowly during this step, so that the drying agent does not clump and possibly entrain the analyte within the drying agent.

- 3.14 Transfer the sample extract by pouring into the drying column. Elute the extract at a 10 mL/min rate into the turbovap vessel. Add 10 mL of hexane to sample bottle and shake. Transfer the hexane wash by pouring into the drying column and elute into the collection vessel. Repeat the rinsing process two more times. Elute the drying column with 60 mL of hexane. Once elution is complete, rinse the column tip with several disposable pipette volumes of hexane.
- 3.15 Turn on the Zymark Turbovap apparatus. Make sure the gas tank valve is on and no pressure registers on the second stage. If pressure registers on the second stage turn off main gas tank valve, place a blank Turbovap vessel into the apparatus, close the lid and bleed the pressure out of the lines. Place

the Turbovap vessel into the Zymark evaporator and close the lid. Slowly increase the pressure on the second stage, while observing the solvent surface for gas flow. Turn up the pressure until the sample is vortexing, but be careful about too high a gas flow which could cause sample splashing.

- 3.16 Set a timer for 10 minutes and check the evaporation process at that time. Increase the gas flow as needed to keep a vortex condition in the sample. Set a timer for 20 minutes and check the sample at that time. Evaporate the sample to a volume of approximately 5 mL (do not go to dryness or loss of PCBs can occur.)
- evaporation unit. Quantitatively transfer the sample extract to a 20 mL vial with three 2 mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the 20 mL vial.
- 3.18 Place the vial with sample extract into the nitrogen blowdown apparatus (45°C) and turn on the gas flow to evaporate the solvent with a gentle stream of purified nitrogen. Evaporate the extract

to a volume of 2 mL (<u>Do Not Go To Dryness - PCB</u>

<u>Loss Will Occur</u>). Add 2 to 4 mL of hexane to the sample and reduce the volume down to 2 mL. Repeat this solvent exchange step two more times.

- 3.19 To the extract add a small amount of mercury, cap the sample and shake for 30 minutes on a wrist action mechanical shaker. This step will remove any sulfur co-extracted from the water sample that can interfere with sample analysis.
- 3.20 Centrifuge the sample for 4 minutes using a bench top clinical centrifuge set to speed number four. Remove sample from centrifuge and transfer extract to a pre-rinsed vial. Rinse the vial containing the mercury three times with 2 mL of hexane, transferring each rinse to the new sample vial. Place the sample on the nitrogen blowdown apparatus and evaporate the extract to 2 mL.
- 3.21 Place the sample extract into the freezer for 5 minutes to chill the sample. Remove the sample from the freezer and add 2 mL of hexane washed sulfuric acid to the sample and shake vigorously for 1 minute. Centrifuge the sample extract for 4 minutes using a bench top clinical centrifuge on

speed setting number 4. Transfer the sample extract (upper phase) into a pre-rinsed vial. Add 2 mL of hexane to the vial containing the sulfuric acid, shake vigorously for 1 minute, centrifuge, and transfer the hexane to the extract vial. Repeat this washing sequence 2 more times and combine the extracts. Place the sample on the nitrogen blowdown apparatus and evaporate the extract to a volume of 2 mL.

- 3.22 The florisil chromatography separates PCBs and certain pesticides (i.e., DDT and analogs) from other co-extracted, polar compounds. The procedure outlined below is for collection of the fraction that contains PCBs and certain organochlorine pesticides only. Additional fractions, eluted by stronger polarity solvent mixtures can be collected if other pesticides are to be determined.
- 3.23 Prepare a micro florisil column in the following manner. Cut the top of a 5 mL disposable pipette at approximately 1 inch from the end of the pipette. Insert a small plug of silanized glass wool into the pipette and position at the 4.5 mL mark. Dry pack the 5 mL pipette with 100% deactivated Florisil to make a column of 1 mL in

height. Make sure the Florisil is well settled by tapping the column with a spatula. Rinse the micro column with 10 mL of hexane and after elution of the 10 mL, rinse the outside tip of the pipette column, with 1 mL hexane. Place a 20 mL vial under the micro column for collection of the elute.

- 3.24 Quantitatively transfer the sample extract to the Florisil micro column and rinse the sample container with 3 successive 0.5 mL hexane volumes. Rinse the disposable transfer pipette into the micro column with 0.5 mL of hexane. Carefully rinse the micro column with 1 mL of hexane and then elute the sample to a total volume of 20 mL with hexane. After collection of the sample rinse the outside tip of the micro column into the collection vial.
- 3.25 Evaporate the solvent with a gentle stream of purified nitrogen gas using the blow down apparatus. Gentle heating can be applied (40°C) to facilitate solvent evaporation. Evaporate the extract to 1.0 mL volume. Never allow the extract to go to dryness (PCB losses can occur).

- 3.26 Quantitatively transfer the extract to a 5 mL volumetric flask using hexane to set to volume. Add 4.5 µL of internal standard (202.0 ppm of octachloronaphthalene in toluene), mix well and transfer to a 10 mL vial. Store extract in freezer until analysis by gas chromatography.
- 3.27 Refer to standard operating procedure NEA608CAP for details on the following:
 - 1. GC packed column screening protocol
 - 2. GC capillary protocol
 - 3. Calculations of PCB concentration
 - 4. QA/QC requirements

ANALYTICAL PROCEDURES

Standard Operating Procedure : NEA608CAP.SOP

NORTHEAST ANALYTICAL, INC. 301 NOTT STREET SCHENECTADY, NEW YORK 12305 (518) 346-4592

STANDARD OPERATING PROCEDURE

LABORATORY METHOD NEA-608CAP

REVISION 3 (6/90)

TABLE OF CONTENTS

		Page Number
1.0	Scope	1
2.0	Summary of Method	2
3.0	Interference	2
4.0	Sample Archiving	3
5.0	Equipment and Apparatus	3
6.0	Reagents	4
7.0	Procedure	7
8.0	Quality Control	21
9.0	References	25

Lab Method NEA-608CAP

Congener-Specific Polychlorinated Biphenyl (PCB) Analysis

Method for Congener-Specific Polychlorinated Biphenyl (PCB)
Quantification and Identification by Capillary Column/Gas
Chromatography with Electron Capture Detection

1.0 Scope

- 1.1 This method is applicable in the determination and quantification of Polychlorinated Biphenyls (PCB) in sediments and soils. This method is a congener-specific determination, employing a high resolution fused-silica capillary chromatographic column. The method includes guidelines set forth in the document: "Quality Assurance Plan, Green Bay Mass Balance Study, I. PCBs and Dieldrin, US EPA Great Lakes National Program Office", prepared by Deborah L. Swackhamer, Quality Assurance Coordinator, Field and Analytical Methods Committees, University of Minnesota, December 11, 1987. This document outlines quality assurance and quality control procedures to be followed by laboratories participating in the Green Bay Mass Balance Study. Where applicable, Northeast Analytical, Inc., will incorporate and utilize this information in quality control of data generated. Instrumental analysis and conditions (Mullin, M.D., 1985, PCB Workshop, US EPA Large Lakes Research Station, Grosse IIe, MI, June.) cited in the Green Bay Mass Balance Study document will be refined to be applicable to an in-house data management software package.
- 1.2 This method will be applied specifically to the determination of PCBs by congener, in sediments. The results of these analyses will be used in studies related to biodegradation of PCBs. Highly sensitive techniques will generally not be required, since samples will contain appreciable quantities

of PCBs. Capillary column methods will be used to effectively separate up to 117 or more peaks representing 187 PCB congeners.

2.0 Summary of Method

- 2.1 This method provides detailed instructions for gas chromatographic conditions, sample extraction, and sample clean-up techniques for analysis of PCBs by capillary gas chromatography.
- 2.2 This method utilizes a mixed Aroclor standard (1232/1248/1262 in the ratio of 25:18:18) for calibration. Method detection limit and practical quantitation limit will be established experimentally using the procedure in USEPA 40 CFR, Part 136, Appx. B.
- 2.3 In general, samples are first extracted with a pesticide-grade solvent. The extracts are further processed by concentrating or diluting, depending on the concentration of PCB, and carried through a series of clean-up techniques. The sample is then analyzed by direct liquid injection onto the gas chromatographic column and detected by an electron capture detector. This method should be performed by a skilled chemist or by an analyst trained in the quantification of trace organics by gas chromatography.
- 2.4 A key component of this method is the importance placed on the chromatographic separation that must be achieved for this congener specific technique. A total of 117 chromatographic peaks are detected, containing 187 PCB congeners in various ratios. This allows an almost complete profile of environmentally occurring PCBs.
- 2.5 Safe laboratory practices should be followed by the analyst at all times when conducting work in the lab. The analyst should refer to the reference file of material safety data sheets to familiarize himself with the hazards of handling the compounds used for standards and samples themselves.

3.0 Interference

- 3.1 One of the major sources of interference in the analysis of PCBs is that organochlorine pesticides are coextracted from the samples. A few of these ECD responding pesticides can be separated cleanly from the PCB profile by the resolving characteristics of the capillary column. Several of the commonly found pesticides and degradation products (DDT, DDE, DDD) overlap the PCB profile envelope and co-elute with several of the minor PCB congeners found in environmental samples. The analyst must be careful in chromatographic pattern review and flag peaks that are suspected of being contaminated so that they are not included in total PCB values generated.
- 3.2 There are several clean-up protocols available that can be used to fractionate PCBs from organochlorine pesticides found in environmental samples. These techniques and there effectiveness will be described in the

sample preparation and clean-up section of this manual. Other separation and clean-up techniques will be included in the section describing procedures to handle samples that contain oil and grease, hydrocarbons, and elemental sulfur.

3.3 Laboratory contamination can occur by introduction of plasticizers (phthalate esters) into the samples through the use of flexible tubing. Samples and extracts should not be exposed to plastic materials. Phthalate esters give response on electron capture detectors, usually as late eluting peaks, and can interfere in PCB quantification.

4.0 Sample Archiving

4.1 Extracts and sediment samples will be retained after analysis. The solvent extracts will be stored in a freezer, while the dry sediment samples can be stored at room temperature, protected from the light.

5.0 Equipment and Apparatus

- 5.1 Gas Chromatograph: Complete system for high resolution, capillary column capability and all required accessories. Northeast Analytical, Inc. will use a Varian Model 3400 gas chromatograph, equipped with capillary oncolumn injection (Septum Programmable Injector), temperature programmable oven, Model 8000 automatic sampler, and fast time constant electron capture detector. A data system (Dynamic Solutions, Maxima Workstation) for chromatographic operations and integration of detector signal is interfaced to the gas chromatograph.
 - 5.1.1 Column: The gas chromatograph column to be used for analysis will be a DB-1 (J&W Company), bonded polydimethylsilicone, 30 meter fused silica capillary column with an internal diameter of 0.25 mm and phase coating thickness of 0.25 microns. This column is capable of resolving 117 chromatographic peaks from the full spectrum of all PCB congeners that could be expected in an environmental sample. Refer to Appendix A and Appendix B for a complete description of PCB congeners identified in each GC chromatographic peak and achievable analytical separations.
- 5.2 Chromatograph Data System: A data system for measuring peak height and peak area. A Maxima 820 workstation (Dynamic Solutions), PC-based data system, will be employed to record detector response and digitally store the chromatographic information on the computer hard disk. This system will allow for chromatographic review of data from the gas chromatograph, electronic peak integration for precise calculations, data base structuring of the analytical information, and archival capabilities.

- 5.3 <u>Soxhlet Extractor</u>: Complete system for use in extracting PCBs from soils, and sediments. The soxhlet extractor consists of a water cooled condensor, a 250 mL, or 500 mL round bottom flask, soxhlet repetitive flushing unit, appropriate heating mantle and controller.
- 5.4 <u>Kuderna-Danish (K-D) apparatus</u>: Complete system to evaporate extraction solvent and concentrate sample extract. The K-D apparatus consists of a concentrator tube (10 mL), evaporation flask (500 mL), and 3-ball snyder column.
 - 5.5 Boiling Chips: Solvent extracted, silicon carbide or equivalent.
- 5.6 <u>Volumetric Flasks</u>: 10, 25, 50, and 100 mL, ground-glass stopper. For standards and sample dilutions.
 - 5.7 Microsyringe: 10, 25, 100, 250, 500 uL for standard preparation.
 - 5.8 Vials: Glass, 10, and 20 mL capacity for sample extracts.
 - 5.9 Bottles: Glass, 50 and 100 mL capacity for sample storage.
- 5.10 <u>Sample Concentrator</u>: An evaporator unit that utilizes a stream of purified nitrogen gas to gently evaporate solvent from samples.

6.0 Reagents

- 6.1 <u>Solvents</u>: Pesticide grade or nano-grade quality. Hexane, acetone, toluene, methylene chloride, methanol, diethyl ether, dimethylformamide, and pentane.
 - 6.2 Magnesium Sulfate: Anhydrous, suitable for pesticide analysis.
 - 6.3 Sodium sulfate: Anhydrous, suitable for pesticide analysis.
 - 6.4 Sulfuric acid: Concentrated, ACS reagent grade.
 - 6.5 Mercury: Triple distilled.
- 6.6 <u>Magnesium silicate (Florisil)</u>: 60-100 mesh, activated at 650 degrees C., reagent grade suitable for pesticide analysis.
 - 6.7 Glass Wool: Silane treated, solvent washed to remove impurities.
- 6.8 Silica Gel: 100-200 mesh, grade 923, Aldrich, deactivated to 7.5% with water.
 - 6.9 Potassium Hydroxide: Pellets, reagent grade.
- 6.10 Octachloronaphthalene: Obtained from Ultra Scientific (Hope, RI), with a purity greater than 95%.

6.11 <u>Polychlorinated biphenyls</u>: Neet commercial material for standard preparation. These materials are multi-component mixtures of PCB congeners and are the actual materials that were used in products such as transformers and capacitors. Monsanto was the largest producer of PCB formulations and sold them under the tradename Aroclor. These standards should be compared to EPA PCB reference materials to verify commercial materials, to be used as standards, have the same pattern and congener distribution.

6.12 Stock Standard Solutions:

- 6.12.1 Stock standards are prepared from individual Aroclor formulations by weighing approximately 0.025 g to the nearest 0.1 mg, and dissolving and diluting to volume in a 25 mL volumetric flask. This will give a stock concentration of 1,000 ppm.
- 6.12.2 The stock standard is transferred into screw-cap vials and stored in a freezer, protected from light. Stock standards should be checked at frequent intervals for signs of evaporation, especially just prior to preparing calibration standards.
- 6.12.3 Stock PCB standards must be replaced after one year, or sooner if comparison with EPA certified check standards indicate a problem.
- 6.12.4 Stock standards for the following are prepared by the above procedure:

Aroclor 1232 Aroclor 1248 Aroclor 1262

- 6.13 <u>Calibration Standard</u>: The calibration standard is prepared by combining Aroclor 1232, Aroclor 1248, and Aroclor 1262 in a 25:18:18 ratio with a final mixture concentration of 2.5 ug/mL, 1.8 ug/mL, and 1.8 ug/mL respectively (total = 6.1 ug/mL). The final concentration of the mixed standard may vary to accommodate instrument sensitivity or more closely represent sample concentrations, but the same ratio values must be maintained. These ratios are strictly maintained so that the percent composition data remains applicable, since it was developed for use under these fixed mixture parameters.
 - 6.13.1 Using a 500 uL microsyringe, accurately add 0.25 mL of stock Aroclor 1232 standard (1,000 ppm) to a 100 mL volumetric flask. Add 0.18 mL of stock Aroclor 1248 and Aroclor 1262 to the same 100 mL of stock volumetric flask. Add the internal standard to the 100 mL volumetric flask as outlined in Section 6.14, and make volume to the 100 mL mark with hexane.
 - 6.13.2 Store calibration standard in the refrigerator at 4 degrees C. in a tightly capped bottle. Calibration standards must be replaced

after six months, or sooner, if comparison with EPA check standards indicate a problem.

- Since the instrument calibration is based on a single point 6.13.3 calibration standard, concentration of PCBs in the sample extracts mustbe within a factor of live of the calibration standard value. Sample extracts that fall outside this range should be diluted or concentrated to be within the accepted concentration range. To facilitate in meeting the established concentration range for samples, sample extracts are to be screened by packed column gas chromatography with electron capture detection to determine their approximate concentrations. Sample extract solution concentrations for this screening procedure will be calculated by using a mixed Aroclor standard (Aroclor 1242 and 1260) with instrument calibration based on the peak weight percent method of Webb and This concentration data is necessary so that the extract concentration can be adjusted to match (within a factor of five) the concentration of the three Aroclor calibration mixture. Besides assessing the PCB extract concentration, the packed column screen will supply information on possible contamination and interfering co-extractables which would indicate further sample clean-up is necessary.
- 6.14 Internal Standard: The internal standard used for capillary gas chromatography of PCBs will be octachloronaphthalene (OCN). Weigh, to the nearest 0.1 mg, solvent, quantitatively transfer the OCN using six successive 2 mL washings to a 50 mL volumetric flask. Be sure to rinse the 10 mL vial walls carefully so that all OCN is completely transferred to the 50 mL volumetric flask. Make the solution to volume using toluene and mix the internal standard solution by shaking the flask several times. This will give a concentration of OCN of 200 ppm. Calculate the exact concentration of OCN based on the amount weighed. Carefully transfer the internal standard solution to 10 mL vials, tightly cap, and store in a freezer.
 - 6.14.1 The OCN internal standard is added to all calibration standards, performance check standards, blanks, samples, and QC samples at an amount to give the same concentration as the major PCBs found in standards and samples. In most cases this will be achieved by spiking 9 uL of OCN internal standard solution to 10 mL of standard or sample extract to give an concentration of 0.1818 ppm.
 - 6.14.2 The internal standard will be added to calibration standards, sample extracts, blanks, and QC samples <u>just</u> prior to gas chromatographic analysis. Thus, the internal standard is used as a quantification spiking standard and will eliminate sample injection and instrument variations, but will not correct for analytical losses during sample preparation.
- 6.15 <u>Performance Check Standard</u>: A performance check standard is prepared from quality assurance/quality control standards obtained from the USEPA Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, Ohio. The performance standard is a mixed Aroclor standard of Aroclor 1232, 1248, and 1262 in the fixed ratio used to prepare the calibration standard

and must be strictly adhered to. The performance standard is made to a concentration at the lower end of the range that sample PCB extract concentrations must fall into (a factor of five of the calibration standard).

- 6.15.1 The performance standard is composed of: 0.50 ug/mL Aroclor 1232, 0.36 ug/mL Aroclor 1248, and 0.36 ug/mL Aroclor 1262, a total of 1.22 ug/mL PCB. This mixture is prepared from a concentration of 5,000 ug/mL in isooctane. The USEPA standards are diluted to 50 ug/mL by pipetting 0.5 mL of standard into a 50 mL volumetric flask and diluting to volume with hexane.
- 6.15.2 Using a 500 uL microsyringe transfer 500 uL of 50 ug/mL Aroclor 1232, 360 uL of 50 ug/mL Aroclor 1248, and 360 uL of 50 ug/mL Aroclor 1262 to a 50 mL volumetric flask. Using a 100 uL microsyringe, add 45 uL of OCN internal standard (final concentration of 0.1818 ug/mL). Dilute to volume with hexane and mix well by shaking and inverting flask several times. The prepared performance check solution will contain a total of 1.22 ug/mL PCB (0.5 ug/mL Aroclor 1232, 0.36 ug/mL Aroclor 1248, and 0.36 ug/mL Aroclor 1262).
- 6.15.3 Transfer the performance check standard to 10 mL vials, cap tightly, and store in a freezer. A new performance check standard must be prepared every three months.
- 6.16 Matrix Spike Standard: The matrix spike standard is prepared in the same manner as the calibration standard outlined in Section 6.13. The only change is that the internal standard is not added to the matrix spike standard. The matrix spike standard is used in quality control to determine percent recoveries of the analytical sample procedures. The matrix spike standard is added at a concentration of the same magnitude as the concentrations exhibited by the samples.
 - 6.16.1 Using the Aroclor stock standards, prepare a stock matrix spike standard as follows. Accurately pipette 12.5 mL of 1,000 ug/mL Aroclor 1232, 9.0 mL of 1,000 ug/mL Aroclor 1248, and 9.0 mL of 1,000 ug/mL Aroclor 1262 into a 50 mL volumetric flask. Dilute to 50 mL with hexane, and mix by inverting the flask several times. The matrix spike standard will contain a total of 610.0 ug/mL PCB (250 ug/mL Aroclor 1232, 180 ug/mL Aroclor 1248, and 180 ug/mL Aroclor 1262).
 - 6.16.2 Transfer the matrix spike standard to 10 mL vials, cap tightly, and store in a freezer. A new matrix spike standard must be prepared every six months, or sooner, if comparison with calibration standards indicate a problem.

8/89

7.0 Procedure

7.1 Calibration:

- 7.1.1 <u>Gas Chromatographic Operation Parameters</u>: Establish the gas chromatographic operation parameters as follows:
 - GC Column: DB-1 (J&W, bonded polydimethylsilicone), 30 meters, 0.25 mm internal diameter, 0.25 micron phase coating.
 - Oven Temperature Program: 50 degrees C. for 0.08 min hold time, 50 degrees C. to 220 degrees C. at 6.0 degrees C./min, hold at 220 degrees C. for 32 min.
 - GC Column Velocity: 30 cm/sec, Helium.
 - Detector: Electron Capture Detector (ECD), attenuation 1, range 10, autozero on.
 - Detector Temperature: 250 300 degrees C.
 - Injector Temperature Program: 45 degrees C. for 0.04 min hold time, 45 degrees C. to 250 degrees C. at 250 degrees C./min, hold at 250 degrees C. for 62.0 min.
 - Detector Make-up Gas: 30 mL/min, Nitrogen.
 - Auxiliary Temperature: 300 degrees C.
 - Autosampler: Multi-vial mode, 0.5 uL sample volume, Fast injection rate (4.0 uL/sec.), 0.0 min injection time, 3 purge pulses.
- 7.1.2 <u>Initial GC Calibration</u>: Prior to running samples the system must be calibrated and the system performance must be verified.
 - 7.1.2.1 Establish the gas chromatographic operation parameters outlined in Section 7.1.1 and prepare the calibration standard composed of a mixture of Aroclors 1232, 1248, and 1262 as outlined in Section 6.13.
 - 7.1.2.2 Load the gas chromatograph autosampler with a vial containing the calibration standard, bracketed by 2 wash vials containing acetone and proceed with the chromatographic analysis. During the chromatographic analysis, GC data acquisition should be performed for future peak integration and data manipulation.
 - 7.1.2.3 Our laboratory will use a computer based data acquisition workstation (Dynamic Solutions, Maxima 820 workstation), interfaced to the gas chromatograph. The workstation processes the detector signal, performs an analog to digital conversion, and stores the digitized chromatograms on the computer hard disk. All data

analysis will be done on the computer specialized software package including peak integration, calculating calibration curves/response factors, report generation, chromatogram hardcopies, and archival of data.

7.1.2.4 Calculate the response factor for each separated and identified peak. Appendix A identifies which congener and or congeners compose each resolvable GC peak in the calibration standard, along with the amount that each congener or co-eluting group of congeners are represented in the calibration standard. Throughout this document the IUPAC PCB numbering system will be used for congener identification, unless otherwise stated. Appendix B is an example of an acceptable chromatogram of the calibration standard, along with peak congener labels for cross reference to data in Appendix A. Chromatographic resolution should be sufficient so as to separate congeners 17 and 18 into two peaks with a valley less than half height of congener 17. Response factors are calculated relative to the internal standard by the following equation:

RRF = (Ax/Ais)x(Cis/Cx)

Where: RRF = Relative response factor of congener(s).

Ax = Area of peak for the cogener(s).

Ais = Area of peak for the internal standard.

Cx = Concentration of the cogener(s).

Cis = Concentration of the internal standard.

7.1.2.5 For congeners that are not found in the mixed Aroclor standard and do not have amount values in the Appendix A table, the following calibration scheme will be employed. Response factors for these additional 19 chromatographic peaks will be adapted from the tabulated data of response factors and relative retention times of all the 209 PCB congeners found in Mullin et al, 1984 (see reference 9.10). These relative response factors will be adjusted for the specific data analysis and quantification employed in this document, based on the calculated response factors generated from the mixed Aroclor standard used for instrument calibration.

7.2 On-going Calibration:

7.2.1 <u>Chromatographic Resolution</u>:

7.2.1.1 Chromatographic resolution should be sufficient so as to separate congeners 17 and 18 into two peaks with a valley less than the half height of congener 17. If this separation is not met, install a new GC column or adjust instrument conditions to achieve stated separation.

7.2.2 Response Factors:

7.2.2.1 The relative response factors calculated from the calibration standard will be verified on each working day by analyzing the performance standard, calculating the selected congener concentrations and comparing to their known concentration. A subset of six congeners will be used to verify the relative response factors before samples are processed. The six congeners include:

#6 and #205 - representing low level peaks in standard

#61 and #181 - representing medium level peaks in the standard #44 and #180 - representing high level peaks in the standard.

7.2.3 After the performance standard is analyzed, calculate the amount for these six congeners and compare those values to the known concentrations by the following equation:

Percent Difference = $[Amt(1)-Amt(2)]/Amt(2) \times 100$

Where: Amt(1)= Amount calculated for congener.
Amt(2)= Known amount for congener.

- 7.2.4 A percent difference greater than $\pm 30\%$ for the two low level peaks (#6 and #205) indicates an instrument problem or unacceptable relative response factors. A percent difference greater than $\pm 10\%$ for the medium level (#61 and #185) and high level (#44 and #180) peaks also indicates an instrument problem or unacceptable relative response factors. If any of the evaluation congeners fail to meet the percent difference acceptance criteria, the calibration standard must be reanalyzed and new relative response factors generated.
- 7.2.5 The performance standard must be analyzed again and values calculated using the new relative response factors. If the performance standard fails to meet the percent difference criteria after re-calibration, sample analysis must not proceed until the problem is found and corrected (i.e., GC gas leak, autosampler lines plugged, broken injector liner).

7.3 Sample Preparation:

7.3.1 Soil and Sediment Samples:

7.3.1.1 To provide guidance to the analyst, as much information about the samples received for analysis should be obtained from the generator and included in the sample log-in records. This may provide information on the expected levels of components being determined and whether dilution of the sample will be necessary.

7.3.1.2 Refer to Section 7.1 for gas chromatographic conditions before running any samples. The instrument calibration performance criteria must be met (see Section 7.2).

7.3.1.3 <u>Sample Preparation</u>:

- 7.3.1.3.1 Process the sample by first decanting water from the top of the sediment. Transfer the sample to a clean pyrex tray and remove any sticks, stones, and other foreign material, if present.
- 7.3.1.3.2 Allow the sample to dry at room temperature by placing in a chemical hood for 24 to 48 hours. Frequently mix the drying sample with a spatula and break up any clumps by crushing. The sample is ready for extraction when the sample has reached a free flowing consistency.

7.3.1.4 <u>Extraction</u>:

- 7.3.1.4.1 Weigh 5 to 20 grams of the air dried sample into a tared cellulose extraction thimble (Whatman 33 mm x 94 mm) and record the weight. Add a plug of silanized glass wool to the top of the thimble.
- 7.3.1.4.2 Add 200 mL of 1:1 mixture of Hexane and Acetone (1:1 Hexane/Acetone) to a 250 mL round bottom flask. Add several boiling chips to the round bottom flask.
- 7.3.1.4.3 Turn on the cooling water to the condenser units that will be used to condense the extraction solvent during the soxhlet extraction of the sample.
- 7.3.1.4.4 Place the sample filled thimble into the soxhlet extractor and attach the soxhlet to the 250 mL round bottom flask and condenser unit.
- 7.3.1.4.5 Place the 250 mL round bottom onto a heating mantle and set the controller to a value of 5 to 6. Once the solvent begins to boil, adjust the controller to achieve a solvent flushing action of once every two to three minutes.
- 7.3.1.4.6 Extract the sample overnight for a minimum of 16 hours. Turn the heating mantle off and allow the apparatus to cool to room temperature. Once cool, rinse the water cooled condenser with several disposable pipette volumes of hexane. Separate the condenser from the soxhlet extractor and rinse the ground glass joint with several disposable pipette volumes of hexane.

- 7.3.1.4.7 Move the soxhlet extractor and attached 250 mL round bottom flask into a chemical hood. Flush the remaining solvent from the soxhlet extractor by tipping the unit to facilitate the syphon action. Using forceps, remove the extraction thimble and allow residual solvent to drain into soxhlet chamber. Rinse the soxhlet chamber with several disposable pipette volumes of hexane, adding enough solvent to be able to flush the soxhlet extractor by tipping to start the siphoning action. Disconnect the soxhlet extractor from the 250 mL round bottom flask and rinse the ground glass joint with several disposable pipette volumes of hexane.
- 7.3.1.4.8 Add several fresh boiling chips to the round bottom flask and attach a 3-ball snyder condensing column to the round bottom flask. Prewet the snyder column with 2 mL of hexane.
- 7.3.1.4.9 Using a heating mantle, evaporate the solvent to approximately 5 mL. Make sure gentle boiling is taking place and that the snyder column sections are not becoming flooded with solvent. Once the extract reaches approximately 5 mL (do not go to dryness or loss of PCBs can occur), remove from heat source and quickly rinse the snyder column with 2 mL of hexane. Allow the glassware to cool to room temperature. Loosen the glass joint between the snyder column and 250 mL round bottom flask and rinse the joint into the round bottom flask with 2 mL of hexane.
- 7.3.1.4.10 Quantitatively transfer the sample extract to a 20 mL vial with three 2 mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the 20 mL vial.
- 7.3.1.4.11 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur). This concentration step removes the final traces of acetone, which needs to be removed before clean-up procedures are performed.
- 7.3.1.4.12 At this point the sample can be processed in either of three ways:
- 1) If the sample is known to be free of interferences, the sample extract volume can be adjusted to 10 mL and and screened by the GC packed column qualitative analysis to determine PCB concentration and necessary sample extract dilution or concentration for final GC capillary quantification analysis.

- If the exact and nature of interfering co-extractables in the sample is unknown, the sample must be analyzed by the packed column qualitative procedure to appropriate clean-up procedures. The sample extract is adjusted to 10 mL and analyzed by packed chromatography. After review of the chromatogram, the sample extract is processed using applicable clean-up techniques. sample clean-up the extract is re-analyzed by GC packed column chromatography to determine PCB concentration and necessary extract dilution or concentration for final GC capillary quantitative analysis.
- 3) If prior knowledge and information exists on what types of interfering substances will be co-extracted with the PCBs, proceed directly with the necessary clean-up procedures. After sample clean-up, the extract is adjusted to 10 mL and analyzed by GC packed column chromatography to determine PCB concentration for final GC capillary quantitative analysis.
- 7.3.1.4.13 Refer to Section 7.4 for sample extract cleanup procedures.
- 7.3.1.4.14 After the sample extract has been analyzed by the GC packed column qualitative screening protocol, the sample is prepared for final analysis by capillary gas chromatography. Dilute or concentrate the sample extract so that the PCB concentration is within the established concentration range (see Section 6.13.3). Spike this sample extract with the internal standard (OCN) as close to the same level that it exists in the calibration standards (typically 0.1818 ug/mL). Place the sample into an autosampler vial and proceed with the capillary gas chromatography analysis.
- 7.3.1.4.15 Refer to Section 7.5 for the calculations of PCB concentration in the sample.
- 7.3.1.4.16 Section 8.0 outlines the necessary quality control (QC) samples to be included with each sample analysis sequence. QC samples are prepared and analyzed along with the samples being measured, and must meet defined acceptance criteria for the sample data to be valid. The QC data is tabulated or plotted on control charts and archived by the laboratory and is available for inspection by the individual requesting the analysis.

7.4 Sample Clean-up

7.4.1 Most extracts of environmental samples that are to be analyzed for PCBs by gas chromatography with electron capture detection

have co-extracted xenobiotics and other interfering substances that must be removed before accurate quantification can take place.

7.4.2 Not all clean-up procedures need to be performed on every sample and several are sample matrix specific. It is the experience and knowledge of the analyst combined with the sampling site history that should guide the selection of which clean-up procedures are necessary.

7.4.3 Hexane/Dimethylformamide partition:

- 7.4.3.1 This procedure is similar to the hexane/acetonitrile partition procedure, but affords quantitative recover of PCBs. This clean-up technique effectively removes lipids and oils from the sample extracts. When necessary, this procedure should be carried out prior to Florisil clean-up.
- 7.4.3.2 Quantitatively transfer the sample extract to a 30 mL vial and adjust the sample volume to 15 mL with hexane. Extract the sample four times with 5 mL of dimethylformamide saturated with hexane.
- 7.4.3.3 Transfer each dimethylformamide extract to a 150 mL separatory funnel. Rinse the transfer pipette with a 0.5 mL of dimethylformamide saturated with hexane into the separatory funnel. Add 100 mL of water containing 2% sodium chloride to the separatory funnel.
- 7.4.3.4 Extract the dimethylformamide/water mixture with 20 mL of hexane. Allow the phases to separate for 5 minutes after vigorously shaking for 30 seconds.
- 7.4.3.5 Decant the dimethylformamide/water phase (lower) into a 250 mL beaker. Transfer the hexane phase to a column containing 3 inches of anhydrous sodium sulfate and elute hexane containing PCBs into a 250 mL round bottom flask.
- 7.4.3.6 Transfer the dimethylformamide/water fraction back to the 150 mL separatory funnel and extract a second time with 20 mL of hexane. Decant the dimethylformamide/water phase back into the 250 mL beaker and add the 20 mL hexane extract to the anhydrous sodium sulfate drying column.
- 7.4.3.7 Repeat Section 7.4.3.6 for a total of three hexane extractions.
- 7.4.3.8 Rinse the separatory funnel into the sodium sulfate column with several disposable pipette volumes of hexane. Rinse the column with several disposable pipette volumes of hexane. Elute the column with an additional 50 mL of hexane to remove all the PCBs. Finally, rinse the drain tube of the column stopcock into the round bottom.

- 7.4.3.9 Add several boiling chips to the 250 mL round bottom flask and rinse the neck of the round bottom with several disposable pipette volumes of hexane. Attach a 3-ball snyder distilling column to the 205 mL round bottom flask and prewet the snyder column with 2 mL of hexane.
- 7.4.3.10 Using a heating mantle, evaporate the solvent to approximately 5 mL. Make sure gentle boiling is taking place and that the snyder column sections are not becoming flooded with solvent. Once the extract reaches approximately 5 mL (do not go to dryness or loss of PCBs can occur), remove from heat source and quickly rinse the snyder column with 2 mL of hexane. Allow the glassware to cool to room temperature. Loosen the glass joint between the snyder column and 250 mL round bottom flask with 2 mL of hexane.
- 7.4.3.11 Quantitatively transfer the sample extract to a 15 mL vial with three 2 mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the 15 mL vial.
- 7.4.3.12 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).
- 7.4.3.13 Quantitatively transfer the sample to a 10 mL volumetric flask and proceed with the packed column GC analysis.

7.4.4 Sulfuric Acid Wash:

- 7.4.4.1 The concentrated sulfuric acid treatment removes hydrocarbons and other colored biogenic compounds which are co-extracted with the environmental PCB residues. The sulfuric acid wash should be done first if a florisil clean-up is also being applied to the sample.
- 7.4.4.2 Quantitatively transfer the sample extract to a 20 mL vial and adjust the volume to approximately 5 mL.
- 7.4.4.3 Add 2 mL of concentrated sulfuric acid to the sample extract and shake vigorously for 30 seconds. Centrifuge samples on low speed (2) using a bench top centrifuge. Transfer the hexane upper layer to a 20 mL vial.
- 7.4.4.4 Wash the sulfuric acid three times with 2 to 3 mL of hexane. Shake each wash vigorously and centrifuge to aid in phase separation. Transfer all three hexane washes to the 20 mL vial.

Make sure to rinse the transfer pipette with 0.5 mL of hexane into the 20 mL vial.

- 7.4.4.5 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).
- 7.4.4.6 If the sample is to be analyzed at this point, quantitatively transfer the extract to a 10 mL volumetric and proceed with the packed column GC screening analysis.
- 7.4.4.7 If the sample needs further clean-up, proceed to the next clean-up procedure.
- 7.4.4.8 <u>Caution</u> must be taken in performing the sulfuric acid wash when high amounts of lipid (i.e., fish samples) or water remain in the sample extract. These types of samples may produce excessive heat due to the exothermic reaction of the sulfuric acid with these materials. The rise in sample temperature can cause sulfonation of the lower chlorinated PCB congeners and therefore losses of PCB from the sample. If high amounts of lipids and water are suspected, cool the sample in an ice bath. Also cool the concentrated sulfuric acid in an ice bath before addition to the sample. Gently shake the sample extract and keep the sample in the ice bath while treating it with the sulfuric acid.
- 7.4.4.9 The pesticides dieldrin and endrin are destroyed by the sulfuric acid treatment. If these pesticides are to be measured they must be fractionated (i.e., silica column) from the PCBs.

7.4.5 Florisil Adsorption Chromatography:

- 7.4.5.1 The florisil chromatography separates PCBs and certain pesticides (i.e., DDT and analogs) from other co-extracted, polar compounds. The procedure outlined below is for collection of the fraction that contains PCBs and certain organochlorine pesticides only. Additional fractions, eluted by stronger polarity solvent mixtures can be collected if other pesticides are to be determined.
- 7.4.5.2 Adjust the sample extract volume to 0.2 to 0.3 mL, so that the sample is applied to the florisil column in a small chromatographic band.
- 7.4.5.3 Prepare a micro florisil column in the following manner. Cut the top of a 5 mL disposable pipette at approximately 1 inch from the end of the pipette. Insert a small plug of silanized glass wool into the pipette and position at the 9 mL mark. Dry pack the 5 mL pipette with 100% deactivated Florisil to make a column of

- 1 mL in height. Make sure the Florisil is well settled by tapping the column with a spatula. Rinse the micro column with 10 mL of hexane, and after elution of the 10 mL, rinse the outside tip of the pipette column, with 1 mL hexane. Place a 20 mL vial under the micro column for collection of the elute.
- 7.4.5.4 Quantitatively transfer the sample extract to the Florisil micro column and rinse the sample container with 3 successive 0.5 mL hexane volumes. Rinse the disposable transfer pipette into the micro column with 0.5 mL of hexane. Carefully rinse the micro column with 1 mL of hexane and then elute the sample to a total volume of 20 mL with hexane. After collection of the sample rinse the outside tip of the micro column into the collection vial.
- 7.4.5.5 Evaporate the solvent with a gentle stream of purified nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).
- 7.4.5.6 If the sample is to be analyzed at this point, quantitatively transfer the extract to a 10 mL volumetric and proceed with the packed column GC screening analysis.
- 7.4.5.7 If the sample needs further clean-up, proceed to the next clean-up procedure.

7.4.6 Sulfur Removal:

- 7.4.6.1 Elemental sulfur is soluble in the extraction of solvents used for sediment and soil samples. It is almost always found as an interferant in these types of samples. Large amounts of sulfur can cause the electron capture detector to signal saturate for long periods during the elution envelope of PCBs. Even small amounts of sulfur can interfere with PCB measurement by co-eluting as a chromatographic peak with certain PCB congeners.
- 7.4.6.2 Sulfur removal clean-up should be routinely done on soil and sediment samples due to its ubiquitous nature. The sulfur removal should be done prior to sulfuric acid and column chromatography clean-up techniques.
- 7.4.6.3 Quantitatively transfer the sample extract to a 20 mL vial and adjust the volume to approximately 5 mL.
- 7.4.6.4 Add 0.3 to 0.5 mL of elemental mercury to the sample extract and vigorously shake for 1 minute. The sulfur is converted to mercuric sulfide and precipitates out of the sample extract.

- 7.4.6.5 Quantitatively transfer the sample extract to a 20 mL vial by using three successive 1.0 mL hexane rinses. Rinse the disposable transfer pipette into the vial with 0.5 mL of hexane.
- 7.4.6.6 The precipitated sulfur can be removed from the sample by performing a sulfuric acid clean-up or Florisil adsorption column clean-up.
- 7.4.6.7 After removal of the sulfur precipitate by either method listed in section 7.4.6.6, quantitatively transfer the sample extract to a 10 mL volumetric flask and proceed with the packed column GC screening analysis.
- 7.4.6.8 If the sample needs further clean-up, proceed to the next clean-up procedure.

7.4.7 Silica Gel Adsorption Chromatography:

- 7.4.7.1 Co-extracted organochlorine pesticides can interfere with PCB identification and quantification. A separation of PCBs from organochlorine pesticides can be accomplished by silica gel adsorption chromatography, allowing the analysis of both PCBs and pesticides as separate fractions.
- 7.4.7.2 Adjust the sample volume to 0.2 to 0.3 mL with hexane. Prepare a column of 7.5% deactivated silica gel (100-200 mesh, Grade 923, Aldrich), packed with 11.5 g of the silica gel. Rinse the column with 50 mL of methylene chloride followed by 50 mL of pentane. Rinse the stopcock drain tube with 1.0 mL of pentane. Place a 250 mL round bottom under the column to collect the sample eluate.
- 7.4.7.3 Quantitatively transfer the sample to the silica column using three successive 0.5 mL pentane rinses. Rinse the disposable transfer pipette into the column with 0.5 mL pentane. Rinse the column walls with several disposable pipette volumes of pentane. Elute the silica gel column with 50 mL of pentane. Rinse the stopcock drain tube with 1.0 mL of pentane. This fraction will contain the PCBs.
- 7.4.7.4 Place a 50 mL bottle under the silica gel column. Elute the silica gel column with 36 mL of 20% methylene chloride in pentane. This second fraction will contain the organopesticides and polychromatic hydrocarbons. This fraction can be stored in a refrigerator and analyzed at a later date if pesticide determination is requested.
- 7.4.7.5 To the round bottom flask containing the PCB fraction add several boiling chips and rinse the neck of the round bottom flask with several disposable pipette volumes of hexane. Attach a

3-ball snyder distilling column to the 250 mL round bottom flask and prewet the snyder column with 2 mL of hexane.

- 7.4.7.6 Using a heating mantle, evaporate the solvent to approximately 5 mL. Make sure gentle boiling is taking place and that the snyder column sections are not becoming flooded with solvent. Once the extract reaches approximately 5 mL (do not go to dryness or loss of PCBs can occur), remove from heat source and quickly rinse the snyder column with 2 mL of hexane. Allow the glassware to cool to room temperature. Loosen the glass joint between the snyder column and 250 mL round bottom flask and rinse the joint into the round bottom flask with 2 mL of hexane.
- 7.4.7.7 Quantitatively transfer the sample extract to a 15 mL vial with three 2 mL hexane rinses. After the sample has been transferred, rinse the disposable transfer pipette with 0.5 mL of hexane into the 15 mL vial.
- 7.4.7.8 Evaporate the solvent with a gentle stream of nitrogen gas using a Pierce Reacti-therm heating block equipped with a 9 port syringe needle gas manifold. Gentle heating can be applied (40-50 degrees C.) to facilitate solvent evaporation. The extract should be concentrated to 0.2 to 0.3 mL, but never allow extract to go to dryness (PCB losses can occur).
- 7.4.7.9 If the sample is to be analyzed at this point, quantitatively transfer the extract to a 10 mL volumetric and proceed with the packed column GC screening analysis.
- 7.4.7.10 If the sample needs further clean-up, proceed to the next clean-up procedure.

7.5 <u>Calculations</u>:

7.5.1 External Standard Calibration (Packed GC):

7.5.1.1 The packed column GC screening analysis will be done by the external standard calibration technique. Calibration and sample quantification will be performed by a commercial GC software package installed on a personal computer. The GC will be standardized by using Aroclor 1242 and Aroclor 1260. These two Aroclor formulations incorporate most environmental PCBs found in sample extracts and provide a good estimate of PCB amount for final dilution or concentration for capillary analysis. A multi-level calibration curve will be developed based on 1 ppm and 10 ppm standards. Weight percent data (Webb and McCall) will be used to generate standard peak amounts.

7.5.1.2 The calibration curves for each calibrated PCB peak will be calculated using the following formula:

Calibration factor = Amount (ug) of component Total area of analyte peak

The calibration curve will be fitted to the calculated calibration factors by a cubic equation to give the best line for all calibration points.

7.5.2 Sample Calculations (Packed GC)

7.5.2.1 The concentration of each identified PCB peak in a sample will be calculated based on the extract volume (not the sample weight or volume) to supply solution concentration values that show if the extract needs to be diluted or concentrated for final capillary GC analysis. The solution concentration of each standardized PCB peak in a sample is calculated as follows:

Concentration (ug/mL) = (Ax) x (CF)

Where: Ax=Area of peak of interest in sample CF=Calibration factor from peak in standard.

7.5.3 Internal Standard Calibration (Capillary GC)

- 7.5.3.1 The capillary column GC analysis will be done by the internal calibration technique. Calibration and sample quantification will be performed by a commercial GC software package installed on a personal computer. The capillary GC will be standardized by using an Aroclor mixture that encompasses all the possible PCB congeners present in environmental samples. Refer to Section 6.13 for details on the calibration standard and Aroclor ratios.
- 7.5.3.2 Response factors for each separated and identified peak in the standard will be calculated using the following formula:

RRF = (Ax/Ais)x(Cis/Cx)

Where: RRF = Relative response factor of congener(s).

Ax = Area of peak for the congener(s).

Ais = Area of peak for the internal standard.

Cx = Concentration of the congener(s).

Cis = Concentration of the internal standard.

7.5.3.3 For the 19 chromatographic peaks that will utilize response factors from tabulated data found in Mullin et at, 1984 (see Section 7.1.2.5), numbers will be manually entered into the peak calibration table used for sample quantification. All calculations will proceed exactly as outlined above for peaks that

have response factors generated from amount information contained in Appendix A.

7.5.4 Sample Calculations (Capillary GC)

The concentration of each identified PCB peak in a sample will be calculated based on the sample air dried weight.

The sample PCB concentration of each standardized PCB peak is calculated as follows:

Concentration (ng/g) = I(Ax)(Cis)(D)[(Ais)(RRF)(Ws)]

Where:

= Peak area for congener(s) being measured. Ax

Cis = Arrgount of internal standard added to sample extract = Dilution factor, if sample was diluted prior to analysis.

= Peak area of added internal standard.

Relative response factor congener(s) measured, as determined in Section 7.5.3.2.

= Air dried weight of sample.

The dilution factor (D) is based on a final extract volume of 1 mL. Typically, the sample is extracted with about 200 mL which is then cleaned up and concentrated to a final volume of 10 mL. further volume adjustment is required, then D=10. lf further dilutions are required, the D=10 x (dilution #1) x (dilution #2) x...

7.5.5 Data Output and Reporting Format:

Several specialized 7.5.5.1 software routines have developed for high resolution PCB analysis to aid the researcher in understanding and organizing the complex data generated from this extremely detailed analysis. Appendix C contains examples of the sample hard copy format that will be used in reporting sample information.

8.0 Quality Control

8.1 Table 3 lists the Quality Control requirements and required recording format that will be applied to the capillary gas chromatographic analysis of PCBs in soils and sediments.

Table 3 - Quality Control Requirements

QC Sample.	Reporting Format	Frequency
Lab Blank	Tabulation	Daily, or with each sample analysis sequence (up to 20 samples).
Performance check	Tabulation	Performance check sample analyzed prior to each sample analysis sequence (up to 10 samples).
Duplicate Analysis	Tabulation	One duplicate analysis per 10 field samples, or monthly if less than 10 samples/month are analyzed.
Matrix Spike	Tabulation	One matrix spike per 10 field samples.

- 8.1.1 <u>Sample Records</u>: All samples that arrive at the lab should be accompanied by a chain of custody document. The following pertinent information should be documented for all samples:
 - 1) Unique label that identifies sample.
 - 2) Location of sample collection site.
 - 3) Date and time of collection.
 - Project name and/or ID number.
 - 5) Field personnel at sampling.
 - 6) Required analysis.

The sample information will be recorded in the Lab Log Books and each sample will be assigned a unique Lab ID Number. The sample analysis will be archived by computer using the Lab ID Number.

8.1.2 <u>Laboratory Blank</u>: The laboratory blank will monitor and assess if the contamination of excessive interference is occurring from laboratory solvents, reagents, and glassware used in processing samples for analysis. The laboratory blank is taken through the sample extraction and clean-up procedures to include all manipulations exposed to actual samples (required volume of solvents, concentration steps, clean-up procedures, etc.). If the laboratory blank is positive for PCB contamination, the source of the contamination must be traced down and eliminated before samples can be processed and analyzed. If non-PCB contamination occurs that interferes with PCB quantification, it too must be traced down and eliminated before proceeding with sample analysis.

The laboratory blank will consist of sandbox sand, purchased at a local hardware store, which has been baked at 250 degrees C. in a vacuum

oven for 24 to 36 hours and allowed to cool and store in glass containers. This is the same procedure used to prepare material which is supplied to EPA's contract laboratories by EMSL/Cincinnvti, for use as clean solid matrix.

- 8.1.2.1 Samples analyzed after a positive laboratory blank should be considered unreliable. If a laboratory blank is positive for PCBs, the source of contamination shall be located and eliminated. If the contamination occurred during the extraction procedure, the samples will require re-extraction and re-analysis. If the contamination occurred after this step, then re-extraction is not appropriate and the existing extracts will be reanalyzed. Any aliquots of the extracts (i.e., injection vials) which could have become contaminated will be discarded.
- 8.1.3 Performance Check Standard: As outlined in section 7.2, a performance check standard will be analyzed on each working day prior to sample analysis and at an interval of one performance standard per each sample analysis sequence (up to 10 samples). The performance check standard must meet the acceptance criteria established in Section 7.2. If the performance check standard falls to meet the acceptance criteria, the calibration standard must be lanalyzed and new response factors generated.
 - 8.1.3.1 The performance check standard must be analyzed again and compared to the acceptance criteria. If the performance check standard fails to meet the acceptance criteria after recalibration, sample analysis must not proceed until the problem is corrected. A typical analytical sequence is as follows:

1) Performance check standard.

2) Method blank.

3 thru 11) Samples (including duplicates, MS).

12) Performance check standard.

13 thru 21) Samples (including duplicates, MS).

22) Performance check standard.

- 8.1.3.2 All samples that were analyzed after the performance check standard exceeded established criteria must be re-analyzed.
- 8.1.4 <u>Duplicate Analysis</u>: Duplicate analysis of the same sample are performed to assess method precision. The percent relative standard deviation of the two measurements on the sample is calculated on total PCB concentration by the following equation:

RSD=(DUP1 - DUP2)/AVG x 100

Where: RSD = Percent relative standard deviation.

DUP1 = the greater of the measured values.

DUP2 = the lesser of the measured values.

AVG = average of the two analyses.

- The percent relative standard deviation must be less 8.1.4.1 than or equal to 25% if the concentration of PCB in the sample is greater than or equal to 0.5 ppm. The percent relative standard deviation must be less than or equal to 50% if the concentration of the PCB in the sample is less than 0.5 ppm.
- 8.1.5 Matrix Spike: Spiked sample matrix data are analyzed to assess analytical accuracy. Thus the sample is spiked and carried through sample analytical procedures including extraction, clean-up, and GC analysis. Matrix spike duplicates are not required for this project.
 - If a sample matrix previously analyzed to be free of PCB is available, spike this sample with matrix spike standard (see Section 6.16) at a concentration similar to sample concentrations. Extract and analyze this spiked sample following procedures used for actual sample analysis. Calculate the percent recovery of the matrix spike by the following:

 $P = A/T \times 100$

Where:

P=Percent recovery, %.

A=Concentration of analyte in the spiked sample aliquot.

T=Known true value of the spike concentration added to the sample aliquot.

8.1.5.2 If an uncontaminated sample is not available, the matrix spike can be performed on a sample previously analyzed. There must be sufficient sample for re-analysis as a matrix spike and the sample must be homogeneous in PCB distribution for valid data to be produced. Preferably a sample of low level should be used in this case. Spike the sample with the matrix spike standard 6.16) at a concentration similar to concentrations. Extract and analyze this spiked sample following procedures used for actual sample analysis. Calculate the percent recovery of the matrix spike by the following:

 $P=(A-B)/T \times 100$

Where:

P=Percent recovery, %.

A=Concentration of analyte in the spiked sample aliquot.

T=Known true value of the spike concentration added to the sample aliquot.

B=Background concentration of PCB in the unspiked sample

The percent recovery for a spiked matrix sample must be greater than or equal to 70% and less than or equal to 130%, based on the total PCB concentration. If a matrix spike recovery sample does not meet the acceptance criteria the cause must be found and corrected. All data collected on samples after the spiked

recovery sample failed to meet the acceptance criteria should be considered unreliable and samples must be re-extracted and analyzed. If additional sample is unavailable, then the data must be flagged as such.

8.1.6 Retention Time Windows

- 8.1.6.1 The GC system should be checked by the analyst to make sure it is functioning properly before establishing retention time windows. Make three analytical measurements of the standard mixture containing the compounds of interest of a minimum of a 72-hour period.
- 8.1.6.2 Calculate the standard deviation resulting from the variation in retention times for each component from three analytical runs.
- 8.1.6.3 The retention time window is defined as plus or minus three times the standard deviation of the three retention time determinations.
- 8.1.6.4 If the standard deviation for a particular component is zero, substitute the standard deviation of a close eluting compound found in the standard solution.
- 8.1.6.5 Besides using the retention time window to assign peaks for quantification, the analyst should also rely on his experience in pattern recognition of multi-residue sample analysis.
- 8.1.6.6 After the daily performance check standard has been analyzed, establish the <u>daily</u> retention time window for each analyte from the retention time of the standard just analyzed. The daily retention time window equals the absolute retention time of the performance check standard (midpoint of the window for the day) plus or minus three times the standard deviation determined in Section 8.1.6.2.
- 8.1.7 Qualitative Compound Identification/Confirmation: The identification of specific congeners in a PCB mixture is done by comparing the retention time of each peak with the retention times of the peaks in a known standard. The standard, consisting of a specified mixture of three Aroclors (1232, 1248, and 1262), has been completely characterized by workers at EPA's Large Lakes Research Station (see Mullin, 1985). Repeated injections of the standard are used to establish an acceptable "retention time window" for each peak. It should be noted, also, that the experience of the analyst plays a significant role in the recognition of PCB patterns. The Laboratory Director of Northeast Analytical, Inc., Robert Wagner, has over 8 years of experience in performing congener-specific analysis of PCBs. Comparative studies using GC/MS and GC-HECD detector analytical techniques have confirmed the validity of this method.

Potential interferences to the PCB analysis are minimized by a number of mechanisms. These include:

The EC detector is largely specific to chlorinated compounds.

2. Rigorous clean-up steps are performed to remove specific interferants, including pesticides, sulfur, lipids and oils, hydrocarbons and biogenic matter. The analyst will strive to produce an extract which is free of interferences.

9.0 References

- 9.1 US EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants", July, 1988.
- 9.2 Standard Methods for the Examination of Water and Wastewater, 16th Edition, Published by: American Public Health Association, American Water Works Association, Water Pollution Control Federation, 1985.
- 9.3 US EPA SW-846, 'Test Methods for Evaluating Solid Waste; Volume 1B Laboratory Manual Physical/Chemical Methods', Office of Solid Waste and Emergency Response, 3rd Edition, 1986.
- 9.4 New York State Department of Health, *Environmental Laboratory Approval Program Certification Manual*, Wadsworth Center for Laboratories and Research, 1988.
- 9.5 Mullin, M.D. 1985. PCB Workshop, US EPA Large Lakes Research Station, Grosse IIe, MI, June.
- 9.6 James L. Lake, Communication, Silica Gel S.O.P., US EPA Environmental Research Laboratory, Narragansett, RI, 1989.
- 9.7 G. Seidl, K. Ballschmiter, Isolation of PCBs from Vegetable Oils: Recovery and Efficiency of *Clean-up* Methods, Chemosphere, No. 5, pp 363-366, 1976.
- 9.8 M. Zell, K. Ballschmiter, Baseline Studies of the GLobal Pollution, III. Trace Analysis of Polychlorinated Biphenyls (PCB) by ECD Glass Capillary Gas Chromatography in Environmental Samples of Different Trophic Levels, Fresenius Z. Anal. Chem., 304, 337-349, 1980.
- 9.9 R.G. Webb, A.C. McCall, Quantitative PCB Standards for Electron Capture Gas Chromatography, J. CHem. Sci., Vol. II, 366-373, 1973.
- 9.10 M.D. Mullin, C.M. Pochini, S. McCrindle, M. Romkes, S.H. Save, 'High-Resolution PCB Analysis: Synthesis and Chromatographic Properties of All 209 PCB Congeners', Environ. Sci. Technol., Vol. 18, No. 6, pp.468-476, 1984.

Appendix A

Congener Composition of Aroclor Calibration Mixture (6.1 ppm)
(Aroclors 1232, 1248, 1262 in a ratio of 25:18:18)

Peak <u>Number</u>	RRT	Amount (ng/mL)	Congener ID
1	0.3243	430	001
2	0.3536	*	001
3	0.3540	260	002
4	0.3685	2.8	003
5	0.3887	22	004, 010
6	0.3941	42	007, 009 006
7	0.3980	500	
8	0.4102	*	005, 008
9	0.4106	10	014
10	0.4133	*	019 030
11	0.4220	*	011
12	0.4245	9.2	
13	0.4269	130	
14	0.4285	74	015, 018 017
15	0.4339	8.8	024, 027
16	0.4389	131	016, 032
17	0.4436	*	023
.18	0.4485	*	034, 054
19	0.4495	1.8	029
20	0.4517	23	026
21	0.4530	10	025
22	0.4564	166	031
23	0.4573	214.	028, 050
24	0.4631	168.5	020, 021, 033, 053
25	0.4670	116.7	022, 051
26	0.4707	27	045
27	0.4746	#	036
28	0.4753	14	046
29	0.4790	*	039 .
30 31	0.4798	129.1	043, 052, 073
32	0.4825	90	049
33	0.4844	50	047
	0.4854	40	048, 075
34	0.4897	*	062, 065
35	0.4915	*	035
36	0.4915	150	044, 104
37	0.4936	88	037, 042, 059
38	0.4990	163	041, 064, 071, 072
39	0.5026	*	068
40	0.5041	*	096
4 1 4 2	0.5031	33	040
43	0.5085	*	057, 103
43	0.5094	5	067, 100

	Peak	RRT	Amauna	_
	Number		Amount (ng/mL)	Congener ID
	44			10
	45	0.5119	7.4	058, 063
	46	0.5145	81	074, 094
		0.5164	210	061 070 074
	47	0.5182	272	061, 070, 076
	48	0.5227	14	066, 093, 095
•	49	0.5274	180	055, 091, 098
	50	0.5308	43	056, 060
	51	0.5324	3	084, 092, 155
	52	0.5346		089
	53	0.5337	48	090, 101
	54	0.5421	23	099
	5.5	0.5437	1.8	112, 119, 150
	5 6	0.5472	3.6	083, 109
	57	0.5500	19	086, 097, 152
	5 8		33.2	081, 087, 111, 115
	59	0.5528	21	085, 116
•	60	0.5549	14	136
		0.5570	71	077, 110
	61	0.5630	*	154
	62	0.5637	13	082
	63	0.5688	57	151
	64	0.5714	22	
	65	0.5727	2.23	124. 135
	66	0.5739	3.3	144
	67	0.5770	* .	107, 108, 147
	68	0.5775		123
	69	0.5833	145	106, 118, 149
	70		*	139, 140
	71	0.5858	8.5	114, 134, 143
	72	0.5875	0.91	122, 131, 133, 142
	73	0.5945	16	146, 161
	74	0.5975	68.04	105, 132
	75	0.6001	147.96	153
	76	0.6100	*	168
	77	0.6106	5 2	141
		0.6121	54.6	179
	78	0.6163	2.5	130
	79	0.6193	13.88	
	80	0.6237	98	137, 176
	8 1	0.6274	12	138, 160, 163, 164
	82	0.6315	3	158
	83	0.6379	34	129
	84	0.6430	*	178
	8 5	0.6439		166
	8 6	0.6468	6	175
	87		150	182, 187
	88	0.6497	4.7	128
	89	0.6529	77	183
	90	0.6583	1.1	167
	91	0.6651	22	185
		0.6724	110	174, 181
	92	0.6778	57	177
	93	0.6838	36.9	156, 171
				4J0. 1/1

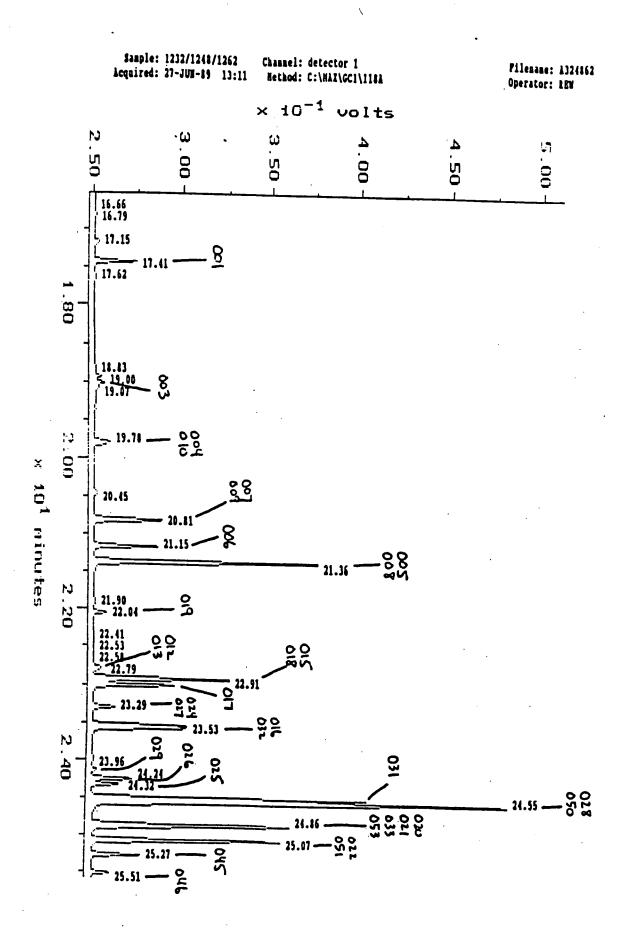
Peak <u>Number</u>	RRT	Amount (ng/mL)	Congener ID	
94	0.6889	2 21		
9 5	0.6919	3.31 *	202	
96			157	
97	0.6928	1.273	173	
98	0.6996	20.697	200, 204	
99	0.7045	19.2	172, 192	
	0.7104	2.18	197	
100	0.7141	240	180	
101	0.7190	14	193	
102	0.7251	4.5	191	
103	0.7323	10	199	
104	0.7554	91.1	170	
105	0.7611	29.9	190	
106	0.7677	0.4	169	
107	0.7779	6.7	198	
108	0.7826	150	201	
109	0.7036	170	196,203	
110	0.8171	1.8	189	
111	. 0.8484	55.9	195	
112	0.8583	24.9		
113	0.8774	4.8	208	
114	0.9058	69	207	
115	0.9241		194	
116	1.0271	4	205	
117		42	206	
* * *	1.0838	0.95	209	

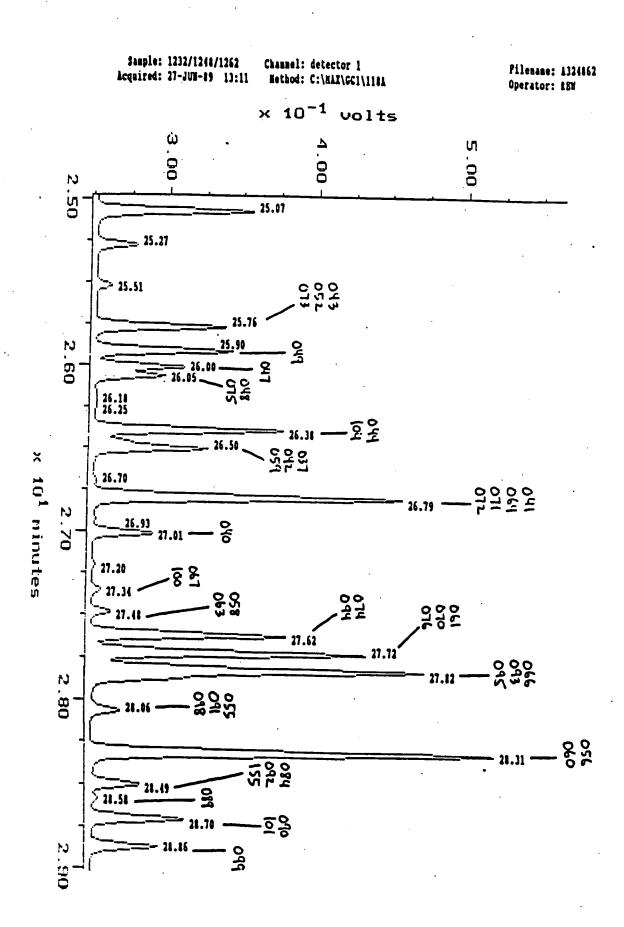
RRT= Relative Retention Time to internal standard OCN= 1.0000

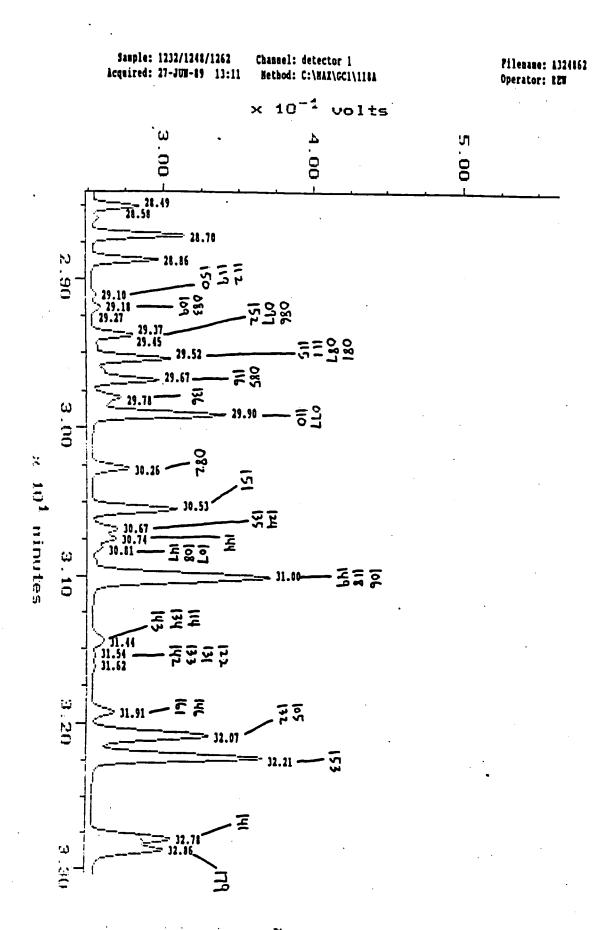
^{*} Refer to Section 7.125 for description of peak quantification for congeners with no amount values.

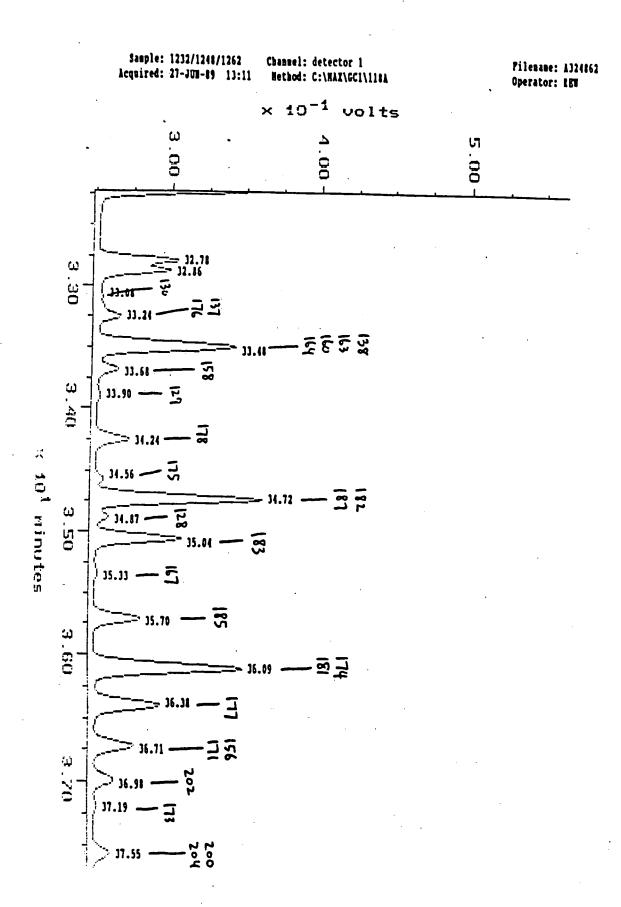
Appendix B

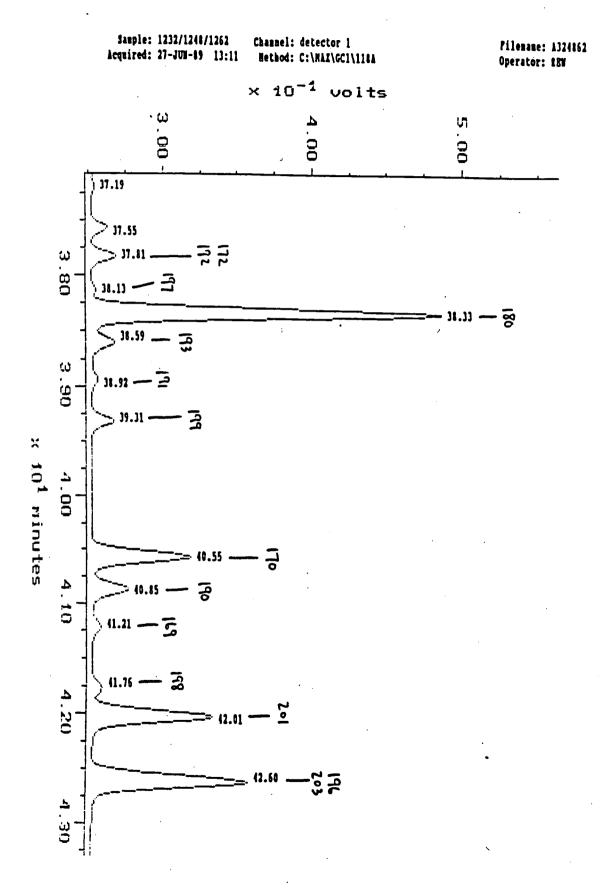
DB-1 Capillary GC Chromatogram of Aroclor Mixture (Aroclors 1232, 1248, 1262 in a ratio of 25:18:18)



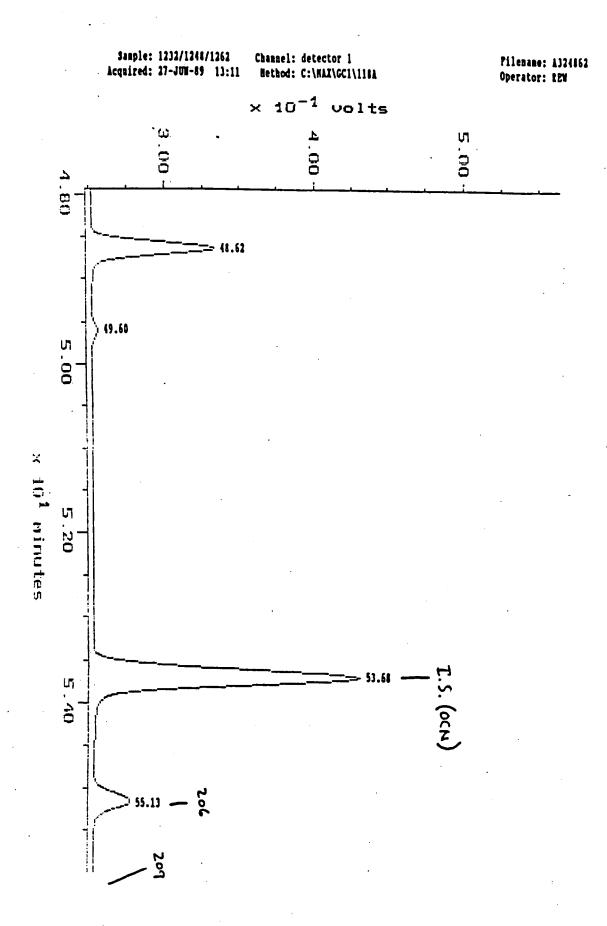








Sample: 1232/1248/1262 Acquired: 27-JUN-89 13:11 Channel: detector 1
Hethod: C:\HAX\GCI\118A Pilemane: 1324862 Operator: 189 × 10⁻¹ volts 43.86 -4.50 \times 10 1 minutes 4.60 4.80



Appendix C

Format of sample reports and specialized data handling and data reduction reports.

MAXIMA 820 CUSTON REPORT

Printed: 27-JUL-1989 15:31:58

SAMPLE SEDIMENT, SECTION 617

47 is Method: HIGH ERS AROCLOR QUANT.

ACQUIRED: 20-JUL-1989 10:34

tate: 5.0 points/sec Duration: 70.000 minutes

Operator: REW

TYPE: UNKN

Instrument: GC2 PILEMANE: 19001

ladez: Disk

Dilution: 4.167

Amount: 0.500

DETECTOR: detector 1

ID	P.E.F	RETENTION TIME (minutes)	PEAR NAME (chlorine no.)	Peak Area	AMOUNT SOULTION (ug/ml)	SAMPLE AMOUNT
1	4	17.41	2 (1)	16822.5	0.052630*	***************************************
5	7	19.89	5 (4,10)	94056.9		0.43862
6	•	20.11	6 (7,9)	4289.9	0.164038!	1.36709!
7	10	21.24	7 (6)	9915.7	0.001725	0.01438
1	11	21.45	1 (5,1)	45470.5	0.004179	0.03483
10	12	22.13	10 (19)	59987.6	0.020305	0.16922
14	13	23.02	14 (15,18)	21477.3	0.027093	0.22579
15	14	23.09	15 (17)	13372.2	0.011548	0.09624
16	15	23.38	16 (24,27)	62691.7	0.005714	0.04762
17	16 .	23.66	17 (16,32)	31481.6	0.013882	0.11569
19	17	24.06	19 (34,54)	5020.4	0.007599	0.06333
21	18	24.34	21 (26)	27177.8	0.001119*	0.00933*
22	19	24.42	22 (25)	21079.9	0.005058	0.04215
23	20	24.60	2) (31)	34627.1	0.004588	0.03824
24	21	24.64	24 [28,50]	37269.8	0.006847	0.05706
25	22	24.99	25(20,21,33,53)	37185.8	0.009023	0.07520
26	23	25.16	26 [22,51]	28349.4	0.008428	0.07024
27	24	25.37	27 (45)	4313.7	0.003843	0.03203
29	25	25.61	29 (46)	3220.1	0.001029	0.00858
J1	26	25.86	31 (52,73)	54018.5	0.001429	0.01191
32	27	26.01	32 (49)	71918.7	0.015520	0.12935
33	28	26.10	33 (47)	98758.1	0.015636	0.13031
37	29	26.49	37 (44,104)	20673.3	0.017085	0.14239
38	30	26.60	38 (37,42,59)	23516.2	0.003803	0.03169
39	32	26.19	39(41,64,71,72)	37428.5	0.005290	0.04409
41	33	27.03	41 (96)	8237.6	0.00480)	0.04003
42	34	27.11	42 [40]	2516.7	0.002223*	0.018531
43	35	27.31	43 (57,103)	11719.7	0.000407	0.00339
11	36	27.49	44 (67,100)	10848.3	0.002115	0.01763
45	37	27.59	45 (51,63)	3222.5	0.001987	0.01656
46	38	27.72	46 (74,94)	13739.7	0.000525*	0.00437
41	40	27.97	48 (66,93,95)	35057.2	0.001839	0.01533
49	41	28.17	49 (55,91,98)	38865.8	0.005087	0.04239
50	42	28.42	50 (56,60)	10320.4	0.007035	0.05163
51	43	28.62	51 (84,92,155)	61168.2	0.001376	0.01146
53	44	21.12	53 (90,101)	119450.8	0.010703	0.08920
54	45	21.91	54 (99)	66138.0	0.026868	0.22392
55	46	29.23	55(112,119,150)	19896.4	0.011244	0.09371
56	47	· 29.31	56 (83,109)	8803.6	0.002236*	0.01463+
57	48 .	29.50	57 (86,97,152)	18519.2	0.001629	0.01525
51	49	29.66	50 (67,111,115)	27694.5	0.002503	0.02086
59	50	29.41	59 (85,116)	320(3.9	0.003021	0.02518
60	99	54.22	I.S. (OCH)	4566128.9	0.002879 0.202000	0.02399
				20278027.1	0.49676	(.14000

NATION 820 CUSTON REPORT

Printed: 27-JUL-1989 16:35:48

SAMPLE: SEDIMENT, SECTION 617

\$7 in Method: BIGB RBS AROCLOR QUART.

ACQUIRED: 20-JUL-1989 10:34

Rate: 5.0 points/sec Duration: 70.000 minutes.

Operator: 124

TYPE: UNCE Instrument: GC1

PILEMANE: 89001 Index: Disk

Dilution: 4.167 Amount: 0.500

DETECTOR: detector 1

ID#	PEŞ	RETENTION TIME	PEAR NAME (chlorine no.)	Peak Area	AMOUNT SOULTION	SAMPLE AMOUNT
		********	tentotine no.1		(ug/ml)	(ug/g
1	. 51	29.92	60 (136)	49734.7		
2	52	30.04	61 (77,110))	124172.2	0.010919	0.09100
3	53	30.30	62 (154)	13529.7	0.016898	0.14083
1	54	30.40	63 (82)	4784.6	0.002472*	0.02060*
5	55	30.69	64 (151)	131715.2	0.000469	0.00391
6	56	30.82	65 (124,135)	64105.8	0.020118	0.16767
	57	30.96	67 (107,101,147)	98570.7	0.007402	0.06169
10	51	31.17	69 (106, 118, 149)	571825.7	0.014373	0.11979
12	59	31.58	71 (114, 134, 143)	20598.9	0.088853	0.74050
13	60	31.79	72(122,31,33,42	15905.4	0.002379	0.01983
14	61	32.09	73 (146,161)	92894.3	0.001914*	0.01595#
15	62	32.26	74 (105,132)	111195.4	0.01)138	0.10949
16	63	32.39	75 (153)	326473.3	0.009147	0.07623
18	64	32.97	77 (141)	117796.2	0.075866 0.007936	0.63227
21	66	33.36	80 (1)7)	40671.6	0.006651	0.06614
22	67	33.44	81 (176)	32099.3	0.002073*	0.05543
23	61	33.69	82(138,160,3,4)	404734.7	0.060371	0.81728*
24	69	33.11	83 (158)	43052.2	0.003625	0.50313
25	70	J4.11	84 (129)	7992.3	0.000879	0.03021
26	71	34.45	85 (178)	54400.3	0.007313	0.00733
21	72	34.78	87 (175)	12024.6		0.06095
29	73	34.94	BE (182,187)	240682.9	0.002160	0.018004
30	14	35.10	89 (128)	39935.1	0.035214	0.29347
31	75	35.27	90 (183)	134422.3	0.002761	0.02301
32	76	35.57	91 (167)	14693.5	0.018141	0.15118
33	77	35.94	92 (185)	46459.0	0.00297	0.02231
34	78	36.34	93 (174,141)	247191.1	0.003893	0.03245
35	79	36.64	94 (177)	143527.1	0.029164	0.24305
36	10	36.97	95 (156,171)	107088.6	0.01615)	0.14046
31	81	37.24	97 (157)	20714.4	0.009579	0.07913
39	12	37.45	98 (173)	5934.7	0.003220	0.02683
40	13	37.83	99 (200,204)	15595.0	0.00200+	0.001664
41	14	38.09	100 (172,192)	57179.2	0.001545+	0.01288*
43	15	31.62	102 (180)	567316.0	0.005713	0.04820
44	16	38.19	103 (193)	32653.3	0.060907	0.50760
45	87	39.23	104 (191)		0.001589+	0.01324*
46	81	39.62	105 (199)	13896.5	0.000639*	0.005334
47	89	40.11	106 (170)	22381.4	0.0013274	0.01106*
· · · 48	- 90	- 41.11	107 (190)	316544.6	0.023393	0.19496
49	92	42.11	108 (198)	73528.3	0.084853	0.01045
50	93	42.37	109 (201)	. 10419.1	0.000479*	0.00400*
51	94	42.95	110 (196,203)	147374.5	0.011801	0.09835
52	95	44.24		193149.4	0.014378	0.11982
53	96	45.96	111 (189)	11566.4	0.000379*	0.003164
56	97	49.10	112 (195) 115 (194)	\$2128.2	0.005304	0.04420
57	98	50.10		195965.6	0.010702	0.08919
58	99	54.22	116 (205)	7874.8	0.000275	0.00229*
		41108	I.S. (OCN)	4566121.9	0.202000	
TOTAL				9775276.4	A / 42444	**********
				777410.9	0.6200121	5.15718*

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET SCHENECTADY, NY 12305 (518) 346-4592

PCB CONCENER AMOUNT and NANOMOLE REPORT

NEA FILE NAME: 89001.MOL

CUSTOMER:

J. EPA

SAMPLE DESCRIPTION: SEDIMENT, SECTION 617

COMMENT:

SAMPLE SHOWED ENVIRONMENTAL ALITERATION

TYPE FOR MIDED PEAK DECONVOLUTION= \$

PEAK NO.	HOLECULAR WT.	AMOUNT	Nanomoles/g(ml) Sample
2	188.70	0.43862	2.32443
5	223.10	1.36709	6.12770
6	223.10	0.01438	0.06446
7	223.10	0.03483	0.15612
8	223.10	0.16922	0.75849
10	257.50	0.22579	0.87685
14	249.00	0.09624	0.38651
15	257.50	0.04762	0.18493
16	257.50	0.11569	0.44928
17	257.50	0.06333	0.24594
19	267.90	0.00933	0.03483
21	257.50	0.04215	0.16369
22	257.50	0.03824	0.14850
23	257.50	0.05706	0.22159
24	257.50	0.07520	0.29204
25	259.50	0.07024	0.27067
26	258.70	0.03203	0.12381
27	292.00	0.00858	0.02938
29	292.00	0.01191	0.04079
31	292.00	0.12935	0.44298
J2	292.00	0.13031	0.44627
33	292.00	0.14239	0.48764
37	292.00	0.03169	0.10853
38	272.40	0.04409	0.16186
39	292.00	0.04003	0.13709
41	326.40	0.01853	0.05677
42	292.00	0.00339	0.01161
43	298.90	0.01763	0.05898
44	298.90	0.01656	0.05540
45	292.00	0.00437	0.01497
46	292.00	0.01533	0.05250
48	293.50	0.04239	0.14443
49	324.70	0.05863	0.18057
۲n	Ses W	0 01146	0 03925

·					
•					
			,		
53	326.40	0.22392	4 40400		
54	326.40		0.68603		
55		0.09371	0.28710		
55 56	326.40	0.01863	0.05708		
50	326.40	0.01525	0.04672		
57	326.40	0.02086	0.06391		
58	326.40	0.02518	0.07714		
59	326.40	0.02399	0. <i>07</i> 350		
60	. 360,90	0.09100	0.25215		
61	315.80	0.14083	0.44595		
62	360.90	. 0.02060	0.05708		
63	326.40	0.00391	0.01198		
64	360.90	0.16767	0.46459		
65	350.50	0.06169	0.17601		
· 67	336.80	0.11979	0.35567		
· 69	337.50	0.74050	2.19407		
<i>7</i> 1	347.80	0.01983	0.05702		
72	336.80	0.01595	0.04736		
73	360.90	0.10949	0.30338		
74	347.80	0.07623	0.21918		
<i>7</i> 5	360.90	0.63227			
\widetilde{n}	360.90	0.06614	1.75193		
80	360.90	0.05543	0.18326		
81	395.30	0.01728	0.15359		
82	360.90		0.04371		
83		0.50313	1.39410		
83 84	360.90	0.03021	0.08371		
. 05	360.90	0.00733	0.02031		
85 87	395.30	0.06095	0.15419		4
	395.30	0.01800	0.04554		
88	395.30	0.29347	0.74240		
89	360.90	0.02301	0.06376		
90	395.30	0.15118	0.38244		
91	360.90	0.02231	0.06182		
92	394.30	0.03245	0.08230		
93	394.30	0.24305	0.61641		
94	394.30	0.14046	0.35623		
· 95	382.20	0.07983	0.20687		
97	360.90	0.02683	0.07434		
98	395.30	0.00166	0.00420	,	
99	429.80	0.01288	0 . 02997		
100	395.30	0.04820	0.12193		
102	395.30	0.50760	1.28409		
. 103	395.30	0.01324	0.03349		
104	395.30	0.00533	0.01348		
105	429.80	0.01106	0.02573		•
106	395.30	0.19496	0.49320		
107	395.30	0.04045	0.10233		
108	429.80	0.00400	0.00931		
109	429.80	0.09835	0.22883		
110	429.80	0.11982	0.27878		
111	395.30	0.00316	0.00799		
112	429.80	0.04420	0.10284		-
115	429.80	0.08919	0.20752		
116	429.80	0.00229	0.00533		
,		4. 00	4.0033		
		•			
		•			
				·.·	
				•	
		•			

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET SCHENECTADY, NY 12305 (518) 346-4592

CONCENER WEIGHT and MOLE REPORT

NEA FILE NAME: 89001.HOL

CUSTOMER:

J. EPA

SAMPLE DESCRIPTION: SEDDMENT, SECTION 617

SAMPLE SHOWED ENVIRONMENTAL ALTERATION

TYPE FOR MIDED PEAK DECONVOLUTION= S

PEAK	RET. TIME	T-a:0-a			CONCENERS	WEIGHT %	HOLE %
2	17.48	1:1	001	.1544	2 22'; 26 24; 25 23' 23; 24' 22'6 22'5; 44' 22'4 236; 23'6 22'3; 24'6 23'5; 22'66' 23'5 23'4 24'5 244'; 22'46 233'; 234; 22'56'	4.716	7.543
5 .	19.89	2:2	004 010	.2245	22' ; 26	14.697	19.885
6	20.88	2:1	007 009	.2566	24 ; 25	0.155	0.209
7	21.24	2:1	006	.2709	22'	0.374	0.507
8	21.45	2:1	005 008	.2785	23 ; 24'	1.819	2.461
10	22.13		019	.3045	22'6	2.427	2.845
14	23.02		018 015	.3387	22'5 ; 44'	1.035	1.254
15	23.09		017	.3398	22'4	0.512	0.600
16	23.38	3:2	024 027	.3508	236 ; 23'6	1.244	1.458
	23.66		016 032	.3625	22'3 ; 24'6	0.681	0.798
19	24.06	3:1 4:4	034 054	.3800	2'35 ; 22'66'	0.100	0.113
21	24.34	3:1	026	.3911	23'5	0.453	0.531
22	24.42	3:1	025	.3937	23'4	0.411	0.482
23	24.60		031	.4024	24'5	0.613	0.719
2Á	24.64	3:1 4:3	028 050	.4031	244' ; 22'46	0.808	0.948
25	24.99	3:1 4:3	021 033	.4170	233' ; 234 ; 22'56'	0.755	
26	25.16	3:1 4:3	022 051	.4267	234'; 22'46' 22'36' 22'55'; 23'5'6	0.344	
27	25.37	4:3	045	.4334	22'36	0.092	
29	25.61	4:3	046	.4450	22'36'	0.128	0.132
31	25.86	4:2	052 073	.4554	22'55' ; 23'5'6	1.391	1.438
32	20.01	4:2	049 047	.4610	22'45 22'44'	1.401	1.448
33	26.10	4:2	047	.4639	22'44'	1.531	1.582
37	26.49	5:4 4:2	104 044	.4832	22'466' ; 22'35'	0.341	0.352
38		3:0 4:2	037 042		344' ; 22'34' ; 233'6		
39	26.89	4:2	064 071	.4990	23'34; 234'6; 23'4'6+	0.430	
	27.03	.5:4	096	.5057	22'366' 22'33' 22'45'6 ; 233'5	0.199	0.184
	27.11	4:2	040	.5102	22'33'	0.036	0.038
43	27.31	5:3 4:1	103 057	.5155	22'45'6; 233'5	0.190	0.191
44	27.49	5:3 4:1	100 067	.5212	22'44'6 ; 21'4'5	0.178	
45	<i>2</i> 7.59	4:1	058 063	.5267	22'44'6; 23'4'5 233'5'; 234'5 244'5; 22'356'	0.047	
46	27.72	4:1 5:3	074 094	.5340	244'5 ; 22'356'	0.165	
48	Z1.91	4:1 5:3	כניט פפט	.544/	77,44, 1, 27,700 1, 77,70,0	0.436	0.469
49	28.17	5:3 4:1	091 098	.5549	22'34'6 ; 22'3'46 ; 233'4 213'4' · 2144'	0.630	0.586
50	7R 47	⊿•1	uer ueu	FATIA .	21714 • 2744	n 171	0 177

	. 28.82	5:2	101 090	.5814	22'34'5; 22'455' 22'44'5 22'34'66'; 233'56; 23'44'6 22'33'5; 233'46 22'3566'; 22'345; 22'3'45 22'345'; 233'55'; 2344'6 22'344'; 23456? 22'34'66' 33'44'; 233'4'6 22'44'56' 22'34'56'; 23'34'5 233'45; 233'45'; 22'34'56 22'34'56; 23'44'5; 233'45 22'33'56'; 22'34'56'; 2344'5 22'33'56'; 23'44'5; 233'45' 22'33'56'; 22'34'5	2.407	2.226
54	28.98	5:2	099	.5880	22'44'5	1.007	0.932
55	29.23	6:4 5:2	150 112	.5969	22'34'66'; 233'56; 23'44'6	0.200	0.185
56	29.31	5:2	083 109	.6029	22'33'5 ; 233'46	0.164	0.152
57	29.50	6:4 5:2	152 097	.6062	22'3566'; 22'345; 22'3'45	0.224	0.207 0.250
58	29.66	5:2	087 111	.6175	22,742, 1, 2342,23, 1, 2344, 6	0.271	0.239
59	29.81	5:2	085 116	.6ZZA	22'344'; 234567	0.236	ريم.ن 0.818
60	29.92	6:4	136	.0 <i>\to</i> 1	22'31'00'	1 514	1.447
61	30.04	4:0 5:2	0// 110	.0 <i>2</i> 53	33 44 ; 233 4 6	0.221	0.185
62	30.30	6:3	104 -	6463	22 TH 30	0.221	0.039
63	30.40	5:4	151	.0400 6400	26 33 7 22125516	1 803	1.508
64	30.69	0:J 6:3 5:1	135 134	4567	22 333 0	0.663	0.571
65 C	30.82	6:3 5:1	107 109	.0303	22 33 50 ; 2 544 3	1 288	1.154
ព	<i>3</i> 0.90	5:1 6:3	1/0 118	6577	22141516 • 2214415 • 212145	7.961	7.120
69 71	31.17	6:3 5:1	149 110	6796	22 34 3 6 7 22 1456 2 2144 5	0.213	0.185
72	21.30	5.1 6.3	122 131	6871	2'33'45: 22'33'46:22'33'55'+	0.171	0.154
73	31.73	6.2	146 161	.6955	22'14'55' : 233'45'6	1.177	0.984
74	32.05	6.3 5.1	132 105	.7015	22'11'46' : 231'44'	0.820	0.711
75	72.20	6.2	153	.7036	22'44'55'	6.797	5.685
ח	12.37	6.2	141	.7203	22'3455'	0.711	0.595
80	33.36	6:2	137	.7329	22'344'5	0.596	0.498
81	33.50	7:4	176	.7305	22'33'466'	0.186	0.142
82	33.69	6:2	138 163	.7403	22'344'5' : 233'4'56 : +2	5.409	4.524
83	33.88	6:2	158	.7429	233'44'6	0.325	0.272
84	34.11	6:2	129	.7501	22'33'45	0.079	0.066
85	34.45	7:3	178	.7537	22'33'55'6	0.655	0.500
87	34.78	7:3	175	.7611	22'33'45'6	0.194	0.148
88	34.94	7:3	187 182	.7653	22'34'5'6; 23'44'5; 233'45' 22'33'45; 22'33'46; 2344'5 22'33'45; 22'33'46; 23'3'55'+ 22'34'55'; 233'44' 22'44'55' 22'34'5 22'34'5 22'34'4'6 22'33'45'6 22'33'45'6 22'34'56' 22'34'56' 22'34'56' 22'34'56' 22'34'56' 22'34'56' 22'34'56' 22'34'56' 22'33'44'5 22'33'45'6 22'33'45'6 22'33'45'6 22'33'45'6 22'33'45'5 22'33'45'6 22'33'45'5 22'33'45'5 22'33'45'5 22'33'45'5 22'33'45'5 22'33'45'5 22'33'45'6 22'33'45'5 22'33'45'6 22'33'45'6 22'33'45'6 22'33'45'6 22'33'45'6	3.155	2.409
89	35.10	6:2	128	.7761	22'33'44'	0.247	0.207
90	35.27	7:3	183	.7720	22'344'5'6	1.625	1.241
91	35.57	6:1	167	.7814	23'44'55'	0.240	0.201
92	35.94	7:3	185	.7848	22'3455'6	0.349	0.267
93	36.34	7:3	174 181	.7965	22'33'456' ; 22'344'56	2.613	2.000
94	36.64	7:3	177	.8031	22'33'4'56	1.510	1.156
95	36.97	7:3 6:1	171 156	.8105	22'33'44'6 ; 233'44'5	0.858	0.678
97	37.24	6:1	157	.8184	233'44'5'	0.288	0.241
98	37.45	7:3	173	.8152	22'33'456	0.018	0.014 0.097
99	37.83	8:4	200 204	1618.	22'33'45'66' ; 22'344'900'	0.130	0.396
100	38.09	7:2	172 192	.8278	22,31,432, 1, 231,432,0	0.318 5.457	4.167
102	38.62	7:2	180	9107	22"344"33"	0.142	0.109
103	38.89	7:2 7:2	191	.8447	23) 44'5'6	0.057	0.044
104	39.23 39.62	7:2 8:4	199	.8494	22'33'4566'	0.119	0.084
105 106	40.88	7:2	170	.8740	22'33'44'5	2.096	1.600
107	41.18	7:2	190	.8740	233'44'56	0.435	0.332
108	42.11	8:3	198	.8845	22'33'455'6	0.043	0.030
109	42.37	8:3	201	.8875	22'33'4'55'6	1.057	0.743
110	42.95	8:3	196 203	.8935	22'33'44'5'6 ; 22'344'55'6	1.288	0.905
111	44.24	7:1	189	.9142	233'44'55'	0.034	0.026
112	45.96	8:3	195	.9321	22'33'44'56	0.475	0.334
115	49.10	8:2	194	.9620	22'33'44'55'	0.959	0.673
116	50.10	8:2	205	.9678	23)'44'55'6	0.025	0.017
-AV	77127						

CONCENTRATION = 9.302

TOTAL MICROPOLES = 0.0308

AVERAGE MOLECULAR WEIGHT = 301.8

NUMBER OF CALIBRATED PEAKS FOUND= 88

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET SCHENECTADY, NY 12305 (518) 346-4592

PCB SUMMARY REPORT

NEA FILE NAME: 89001.HOM

CUSTOMER:

J. EPA

SAMPLE DESCRIPTION: SEDIMENT, SECTION 617

COMMENT:

SAMPLE SHOWED ENVIRONMENTAL ALTERATION

Total PCBs in Sample= 9.30

PCB Homolog Distribution

Homolog Series	Percent in Sample
"Mono	4.72
Di	17.30
Tri	9.31
Tetra	6.73
Penta	14.97
Hexa	23.29
Hepta	19.58
Octa	4.10
Nona	0.00
Deca	0.00

Ortho Cl / biphenyl Residue = 1.99

Meta + Para Cl / biphenyl Residue = 2.30

TOTAL C1 / biphenyl Residue = 4.29

METHOD PRECISION AND ACCURACY

METHOD DETECTION LIMITS

Northeast Analytical, Inc. Quality Assurance and Quality Control Data

QA/QC data enclosed is divided into four sections outline below.

- I. Precision and Accuracy data for Aroclor 1242 as outlined in 40 CFR, Part 136, App. A, Method 608; 1-July-1985.
- II. Method Detection Limit data for Aroclor 1242 as outlined in 40 CFR, Part 136, App. B; 1-July-1985.
- III. Monthly surrogate recovery chart November 1991, for nonachlorobiphenyl surrogate using separatory funnel extraction. Quality control limits are outlined on chart. Where:

UCL = upper control limit
UWL = upper warning limit
LWL = lower warning limit
UCL = lower control limit

IV. Matrix spike recovery chart for Aroclor 1242. Quality control limits are outlined on chart. Where:

UCL = upper control limit
UWL = upper warning limit
LWL = lower warning limit
UCL = lower control limit

Precision and Accuracy Multicomponent Liquid Table

File Name: q:\qc\p&a\gc1a42

Date:

01 - May - 92

Precision and Accuracy (P&A) calculations and limits are based on procedures outlined in 40 CFR, part 136, App A; Method 608, 1-July-85.

Compound: Aroclor 1242	Analysis: GREEN BAY ANALYSIS
Matrix:H20	Instrument: GC-1 CAPILLARY
Extraction:sep. funnel	Column: DB-1
Spike conc: 25.2 ng/L	

Γ	NEA	Extr	File	Analysis	Expt	Percent	Percent
	Samp	Date	Name	Date	Conc	Recovery	Recovery
	ID_				(ng/L)	(%)	Valid
1	921263	21-Apr-92	921263	28-Apr-92		108.89%	in criteria
2	921264	21-Apr-92	921264	28-Apr-92	23.14	91.83%	in criteria
3		21-Apr-92	921265	28-Apr-92	24.38	96.75%	in criteria
4	921266	21-Apr-92	921266	28-Apr-92	24.65	97.82%	in criteria
				n:	4		

Aroclor 1242
Precision and Accuracy Limits *

s (ug/L) P (%)

12.2 39% 150%

AVG: 24.90
STD (s): 1.81
%RSD: 7.3%
VALID (s): in criteria

Comments:	· · · · · · · · · · · · · · · · · · ·	 · .				
		 			-	
						
		 •				
			•	•		

98.82%

^{*} Limits outlined in 40 CFR Part 136, App. A, Method 608, 1-July-1985

Precision and Accuracy Multicomponent Liquid Table

File Name: q:\qc\p&u\gc2a42

Date:

01-May-92

Precision and Accuracy (P&A) calculations and limits are based on procedures outlined in 40 CFR, part 136, App A; Method 608, 1-July-85.

Compound: Aroclor 1242	Analysis: GREEN BAY ANALYSIS
Matrix:H20	Instrument: GC-2 CAPILLARY
Extraction:sep. funnel	Column: DB-1
Spike conc: 25.2 ng/L	

	NEA	Extr	File	Analysis	Expt	Percent	Percent
1	Samp	Date	Name	Date	Conc	Recovery	Recovery
L	ID				(ng/L)	(%)	Valid [*]
ا ا		21-Apr-92	921263	28-Apr-92	26.83	106.47%	in criteria
2	921264	21-Apr-92	921264	28-Apr-92	24.40	96.83%	in criteria
3 [21-Apr-92	921265	28-Apr-92	24.57	97.50%	in criteria
<u>ا</u> ا	921266	21-Apr-92	921266	28-Apr-92	23.04	91.43%	in criteria
		<u> </u>		n·	4		

Aroclor 1242

Precision and Accuracy Limits *

s (ug/L) P (%)
12.2 39% 150%

AVG: 24.71 98.06%

STD (s): 1.57 %RSD: 6.4% VALID (s): in criteria

Comments:_	<u> </u>							
						-		
		•						
					-			
· ·							 	

^{*} Limits outlined in 40 CFR Part 136, App. A, Method 608, 1-July-1985

Method Detection Limits Multicomponent Liquid Table

 $File \ Name: \ q:\qc\md1\gc1a42$

Date:

01-May-92

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound: A1242	Analysis: GREEN BAY ANALYSIS			
Matrix:H20	Instrument: GC-1 CAPILLARY			
Extraction:sep. funnel	Column: DB-1			
Spike conc: 25.2 ng/L				

	NEA	Extr	File	Analysis	Expt	Percent
	Samp	Date	Name	Date	Conc	Recovery
	ID	· · ·			(ng/L)	(%)
1		21 – Apr – 92	921263	23-Apr-92	27.44	108.89%
2	921264	21-Apr-92	921264	23-Apr-92	23.14	91.83%
3	921265		921265	23-Apr-92	24.38	96.75%
4		21 – Apr – 92		23-Apr-92	24.65	97.82%
5	921267	21 – Apr – 92	921267	23-Apr-92	22.65	89.88%
6		21 – Apr – 92		23-Apr-92	26.39	104.72%
7			921269	23-Apr-92	25.15	99.80%
8	921270	21-Apr-92	921270	23-Apr-92	24.65	97.82%

One sided Student's t values (t)

at the 99% level.

Number (n)	(t) value		
7	7	3.143	
8	3	2.998	

Number (n):	8	
AVG:	24.81	
STD (s):		ng/L
%RSD:	6.3%	
MDL:	4.70	ng/L
DOL.	00.54	L - //

PQL: 23.51 ng/L VALID: valid

MDL calculations:

MDL = t * s

Where:

t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

PQL = MDL * 5

Comments:_	 		 	

Method Detection Limits Multicomponent Liquid Table

File Name: q:\qc\md1\gc2a42

Date:

01-May-92

Method Detection Limit (MDL) calculations as based on procedures outlined in 40 CFR, part 136, App B; 1-July-85.

Compound: A1242	Analysis: GREEN BAY ANALYSIS			
Matrix:H20	Instrument: GC-2 CAPILLARY			
Extraction:sep. funnel	Column: DB-1			
Spike conc: 25.2 ng/L				

ſ	NEA	Extr	File	Analysis	Expt	Percent
	Samp	Date	Name	Date	Conc	Recovery
Į	ID				(ng/L)	(%)
1 [21 - Apr - 92	921263	23-Apr-92	26.83	106.47%
2	921264	21-Apr-92	921264	23-Apr-92	24.40	96.83%
3 [21 – Apr – 92		23-Apr-92	24.57	97.50%
4		21-Apr-92		23-Apr-92	23.04	91.43%
5	921267	21 – Apr – 92	921267	23-Apr-92	23.62	93.73%
6	921268	21 - Apr - 92	921268	23-Apr-92	26.88	106.67%
7 [921269	21-Apr-92	921269	23-Apr-92	26.44	104.92%
8	921270	21-Apr-92	921270	23-Apr-92	25.39	100.75%

Number (n): AVG:

25.15 ng/L 1.47 ng/L

STD (s): %RSD: MDL:

5.9% 4.42 ng/L

8

PQL:

22.11 ng/L VALID: valid

MDL calculations:

at the 99% level.

Number (n)

MDL = t * s

(t) value

3.143

2.998

Where:

One sided Student's t values (t)

8

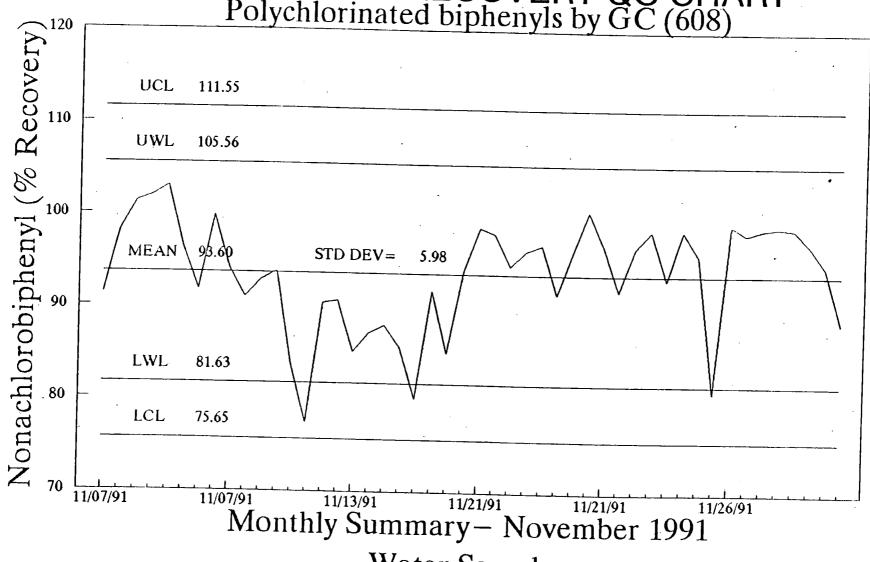
t = one sided Student's t value for the number of replicates at the 99% level

s = standard deviation of the population

PQL calculations:

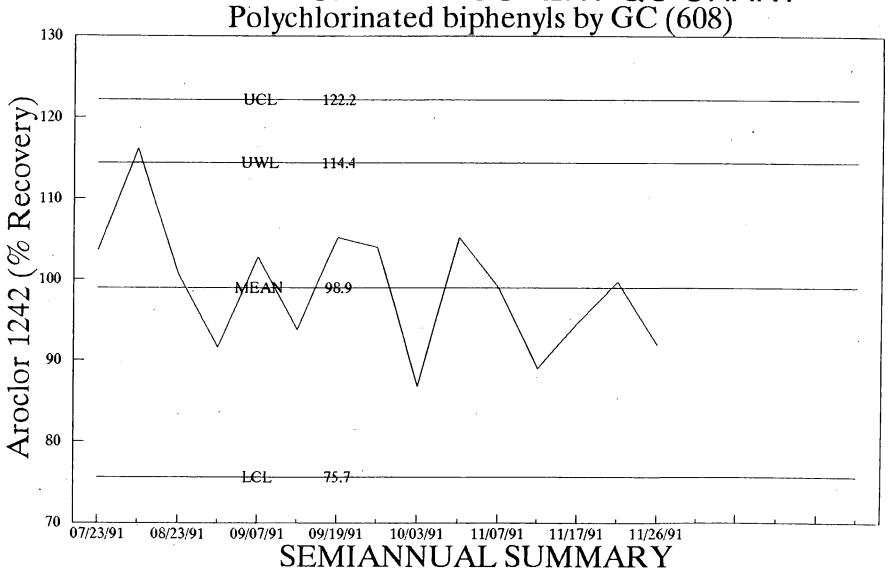
·	Others	il\mdi mi01.wk3 920428;gy
		·
Comments:		
		•

SURROGATE RECOVERY QC CHART Polychlorinated biphenyls by GC (608)



_ Water Samples

MATRIX SPIKE RECOVERY QC CHART



Water Samples

CALIBRATION PROCEDURES AND FREQUENCY

CALIBRATION PROCEDURES AND FREQUENCY

All calibration procedures and frequency for testing instrument calibration for gas chromatographic analysis of PCBs can be found in the standard operating procedure, NEA608CAP, included in this document.

DATA REDUCTION AND REPORTING

DATA REDUCTION AND REPORTING

A computerized chromatography workstation (Maxima, Dynamic Solutions Inc.) is utilized to transform chromatographic signal into peak area counts which is used to calculate concentration based on response factors generated from calibration standards. All pertinent information (ie. sample weight or volume, extract volume, extract dilutions, and internal standard concentration used for quantification) can be entered into workstation methods so that final hardcopy reports sample are on basis. The chromatographic data computer file is kept on the computer network as an active file for six weeks and then migrated to a 1.2 gigabyte tape backup system for long term data archiving. In this manner data can be retrieved at any time in the future.

The workstation ASCII report can be utilized in various spreadsheet and wordprocessing programs to further format or manipulate the data. Specialized software has been developed to analyze and report congener specific PCB chromatographic data and identify Aroclor type by target peak analysis. Identification of Aroclors found in samples is also performed by visual inspection of chromatograms. Experience in reviewing PCB formulations is an important aspect in determining and correctly assigning Aroclor types or recognizing when mixtures exist in samples. Examples of maxima workstation reports an in-house custom reports are included for review.

HAXIMA 820 CUSTOM REPORT

Printed: 29-APK-1992 12:42:30

Type: UNKN

Filename: PCO427A

Index: Disk

SAMPLE: PER CHK 1.22PPM

Acquired: 29-APR-1992 4:23 Rate: 5.0 points/sec

Duration: 70.000 minutes

Operator: REW

DETECTOR: detector 1

101	₽K #	RETENTION TIME {minutes}	PEAK NAME (chlorine no.)	Peak Area	AMOUNT SOULTION (UG/ML (PPM))	SAMPLE AMOUNT (NG/L (PPT))
3	ì	16.91	2 (1)	11849.4	0.086707	86.70676
4	2	18.49	4 (3)	9862.9	0.045561	45.56116
5	. }	19.27	5 (4,10)	27550.6	0.005235	5.23456
Ó	4	20.29	6 (7,9)	77566.2	0.004220	4.21988
7	5	20.64	7 [6]	58501.8	0.008273	8.27265
Ů.	6	20.84	8 (5,8)	245752.8	0.096718	96.71769
10	7	21.52	10 (19)	15653.0	0.002296	2.29608
13	8	22.26	13 (12,13)	10175.8	0.001553	1.55327
14	ý.	22.39	14 (15,18)	212648.4	0.029989	29.98865
15	10	22.47	15 (17)	106388.8	0.016909	16.90874
16	11	22.76	16 (24,27)	26158.7	0.001905	1.90497
17	12	23.00	17 (16,32)	215044.3	0.030675	30.67522
19	13	23.44	19 (34,54)	2336.0	0.000545*	0.54464*
20	14	23.60	20 (29)	3691.4	0.000284	0.28411
21	15	23.72	21 (26)	51247.5	0.005591	5.59117
22	1.6	23.79	22 (25)	30632.0	0.002182	2.18235
23	17	23.98	23 (31)	327423.1	0.033107	33.10746
24	18	24.02	24 (28,50)	507087.2	0.035663	35.66324
35	19	24.33	25(20,21,33,53)	291397.9	0.035954	35.95373
26	20	34.54	26 (22,51)	219466.0	0.024327	24.32701
27	21	24.74	27 (45)	65159.6	0.006212	6.21168
29	22	24.97	29 (46)	25349.0	0.003296	3.29617
11	23	25.23	31 (43,52,73)	228130.6	0.027273	37.27323
32	24	25.37	32 (49)	215705.3	0.017765	17.76477
13	25	25.47	33 (47)	115182.2	0.008897	8.39718
34	26	25.52	34 (48,75)	102709.7	0.008340	8.33977
16	27	25.71	36 (35)	2469.1	0.000749*	0.74935*
37	. 28	25.85	37 (44,104)	268247.5	0.026908	26.90827
38	. 29 -	.25.96	38 (37, 42, 59)	150484.5	0.012881	12.88130
39	30	26.25	39(41,64,71,72)	435944.1	0.027881	27.88062
41	31	26.38	41 (96)	5387.2	0.001520*	1.51951*
42	32	26.47	42 (40)	95434.8	0.007844	
43	33	26.67	43 (57,103)	3829.0	0.000722*	7.34373
+4	34	26.81	14 (67,100)	14689.1	0.001165	0.72213*
45	35	26.94	45 (58,63)	24862.1		1.16500
16	36	37.08	46 (74,94)	241144.8	0.001508	1.50824
÷7	37	27.18	47 (61,70,76)	381150.8	0.013231	13.23103
+8	38 .	27.28	18 (66, 93, 95)	569965.2	0.037123	37.12313
49	39	27.52	19 (55,91,98)		0.048404	18.10368
50	10	27.76	50 (56,60)	51869.5	0.003855	3.85454
51 -	41	27.95	51 (84,92,155)	464603.0	0.029600	29.60043
	••	41174	JI (07,78,133) .	80827.8	0.009481	9.48071

54 55 56 57 58 59 60	43 44 45 46 47 48 49	28.15 28.30 28.52 28.60 28.77 28.92 29.05 50.83	53 (90,101) 54 (99) 55(112,119,150) 56 (83,109) 57 (86,97,152) 58(81,87,111+15 59 (85,116) 1.S. (OCN)	149773.4 83154.5 5239.4 13861.7 59487.1 118751.3 90265.2 5427130.5	0.009591 0.003916 0.000309 0.000769 0.003787 0.006396 0.003398 0.181800#	9.59093 3.91643 0.30865 0.76912 3.78712 6.39587 3.39771
TOTAL				6557909.6	0.7911304	791.129994

Yalue not included in TOTAL calculation.
kesult calculation was based on a manually entered response factor.

HAXIMA 820 COSTON REPORT

Printed: 30-APR-1992 9:18:22

Type: UNKN

Pilename: PC0427A

Index: Disk

SAMPLE: PER CHK 1.23PPM

Acquired: 29-APR-1992 4:23 Rate: 5.0 points/sec

Duration: 70.000 minutes

Operator: REW

DETECTOR: detector 1

ID i	PX#	RETENTION TIME	PEAK NAME (chlorine no.)	Peak Area	NOITJUOZ TRUONÁ (ug/al)	SAMPLE AMOUNT
1	50	29.15	60 (136)	72045.8	0.003158	1 16020
2	51	29.26	61 (77,110))	188812.8	0.012024	3.15830
4	52	29.59	63 (82)	53098.7	0.012024	12.02422
5	. 53	29.85	64 (151)	178930.8	0.013143	2.54258
6	54	29.97	65 (124,135)	51009.2	0.005155	13.14282
7	55	30.04	66 (144)	47102.5	0.00514	5.15509
8	56	30.09	67(107,108,147)	12137.2	0.000514	0.51421
9	57	30.17	68 (123)	4922.9	0.000585*	0.61989
10	58	30.27	69(106,118,149)	424481.7	0.026375	0.58543.
12	59	30.67	71 (114,134,143)	24712.8	0.001470	26.37491
13	61	30.84	72(122,31,33,42	4592.9	0.000150	1.46988
14	62	31.12	73 (146,161)	45654.5		0.15005
15	63 -	31.26	74 (105,132)	210563.3	0.003616	3.61560
16	64	31.40	75 (153)	381756.4	0.012419	12.41926
18	65	31.91	77 (141)	169721.4	0.027596	27.59622
19	66	31.98	78 (179)	164671.1	0.011231	11.23142
20	67	32.20	79 (130)	3569.2	0.013407	13.40686
21	68	32.33	80 (137,176)	61080.5	U.000273	0.27299
22	69	32.55	82(138,160,3,4)	363805.8	0.003097	3.09676
23	70	32.72	83 (158)	47194.4	0.018922	18.92189
24	71	32.93	84 (129)	6295.8	0.002338	2.33847
25	72	33.25	85 (178)	86846.8	0.000417	0.41725
27	73	33.54	87 (175)		0.008197	8.19721
28	74	33.68	88 (182,187)	18470.1	0.001378	1.37815
29	75	33.82	89 (128)	474858.0 -25333.4	0.031137	31.13739
30	76.	33.98	90 (183)	234179.4	0.000799	0.79905
31	17	34.23	91 (167)	4701.6	U. 016451	16.45107
32	78	34.57	92 (185)	111899.6	0.000121	0.12106
33	79	34.93	93 (174,181)		0.005009	5.00863
34	80	35.20	94 (177)	425562.4	0.021877	21.87724
35	81	35.49		198314.7	0.012156	12.15618
36	82	35.73	95 (156,171)	106886.9	0.007710	7.70959
38	83	35.92	96 (202)	62590.6	0.000782	0.78176
39	84	36.26	98 (173)	8250.4	0.000251	0.25069
40	85	36.50	99 (200,204)	49392.7	0.004851	4.85061
41	86	36.79	100 (172,192)	72772.0	0.004379	4.37864
12	87	36.97	101 (197)	11671.3	0.001280	1.27969
43	88	30.97 37.20	102 (180)	1035382.9	0.045622	45.62242
11	89	37.50	103 (193)	79237.9	0.005931	5.93130
45	90	37.86	104 (191)	22795.0	0.001483	1.48277
46	91		105 (199)	73812.4	0.002423	2.42335
70	11	38.98	106 (170)	342516.5	0.019688	19.68775

48 49 .50 51 52 53 54 55 56	94 95 96 97 98 99 100 101 102 103	40.09 40.31 40.84 42.00 43.51 44.01 44.93 46.31 47.19 50.83	108 (198) 109 (201) 110 (196,203) 111 (189) 112 (195) 113 (308) 114 (207) 115 (194) 116 (205) 1.S. (0CN)	28864.4 442694.5 522208.8 9254.7 182451.8 33187.4 25219.4 486428.3 22195.3 5427130.5%	0.001498 0.034368 0.036344 0.000383 0.012153 0.005873 0.001090 0.014759 0.000771 0.1818004	1.49784 34.26822 36.34442 0.38272 12.15317 5.87323 1.09021 14.75931 0.77138
58 59 rotal	103 104 105	52.19 58.09	1.S. (OCN) 117 (206) 118 (209)	5427130.5# 211250.3 3886.0 8039743.5	0.181800¢ 0.010032 0.000208 	10.03201 0.30773

Yalue not included in TOTAL calculation.
 kesult calculation was based on a manually entered response factor.

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET SCHENECTADY, NY 12305 (518) 346-4592

PCB CONGENER AMOUNT REPORT

NEA FILE NAME: 0515BW1.mol

CUSTOMER:

CIBA-GIEGY CORPORATION

SAMPLE DESCRIPTION: 920515BW1E W/B

COMMENT:

CRANSTON WATER COLUMN PROJECT PCB H2O SAMPLES COC:5/14/92

DATE ACQUIRED:

19-MAY-1992 5:24

TYPE FOR MIXED PEAK DECONVOLUTION= S

PEAK NO.	PEAK NO. MOLECULAR WT.		Ferntomoles/g(ml) Sample
l l	154.20	0.00000	0.00000
2	188.70	0.00000	0.00000
3	188.70	0.00000	0.00000
.4	188.70	0.00000	0.00000
.5	223.10	0.00000	0.00000
6	223.10	0.00000	0.00000
7	223.10	0.00000	0.00000
8	223.10	0.00000	0.00000
9	223.10	0.00000	0.00000
10	257.50	0.00000	. 0.00000
11	257.50	0.00000	0.00000
12	223.10	0.00000	0.00000
13	223.10	0.00000	0.00000
14	249.00	0.00000	0.00000
15	257.50	0.00000	0.00000
16	257.50	0.00000	0.00000
17	257.50	0.00000	0.00000
18	257.50	0.00000	0.00000
19	267.90	0.00000	0.00000
20	257.50	0.00000	0.00000
21	257.50	0.00000	0.00000
22	257.50	0.00000	0.00000
23	257.50	0.27008	1.04885
24	257.50	0.18394	0.71433
25	259.50	0.00000	0.00000
26	258.70	0.00000	0.00000
27	292.00	0.00000	0.00000
28	257.54	0.00000	0.00000
29	292.00	0.00000	0.00000
30	257.50	0.00000	0.00000
31	292.00	0.00000	0.0000
32	292.00	0.00000	0.00000

13						
25		2.4	202 (10)	0.00000	0.00000	
36	,					
36		35	292.00	0.00000	0.00000	
37 292,00 0.19508 0.47151 38 772.40 0.00000 0.00000 39 292.00 0.20004 0.66857 40 292,00 0.00000 0.00000 41 1.25.40 0.00000 0.00000 42 2.27.00 0.00000 0.00000 44 2.28.50 0.00000 0.00000 44 2.29.50 0.00000 0.00000 44 2.29.50 0.00000 0.00000 45 292.00 0.00000 0.00000 46 292.00 0.00000 0.00000 47 292.00 0.00000 0.00000 48 293.50 0.49387 1.66259 49 324.70 0.00000 0.00000 50 292.00 0.00000 0.00000 51 326.40 0.00000 0.00000 52 326.40 0.00000 0.00000 53 326.40 0.00000 0.00000 53 326.40 0.00000 0.00000 53 326.40 0.00000 0.00000 54 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 56 326.40 0.00000 0.00000 57 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 50 326.40 0.00000 0.000000 50 326.40 0.000000 0.000000 50 326.40 0.000000 0.000			257 50	0.00000	0.00000	
38						
38		37	292.00	0.19608	0.67151	
39			272.40	0.00000	01.00000	4
40						
40 292.00 0.00000 0.00000 4 11 326.40 0.00000 0.00000 0.00000 42 292.00 0.00000 0.00000 0.00000 43 298.90 0.00000 0.00000 0.00000 44 298.90 0.00000 0.00000 0.00000 45 298.90 0.00000 0.00000 0.00000 46 298.90 0.00000 0.00000 0.00000 47 298.90 0.00000 0.00000 0.00000 48 298.90 0.00000 0.00000 0.00000 49 292.00 0.00000 0.00000 0.00000 50 292.00 0.00000 0.00000 0.00000 51 326.40 0.00000 0.00000 0.00000 52 326.40 0.00000 0.00000 0.00000 53 326.40 0.00000 0.00000 0.00000 55 326.40 0.00000 0.00000 0.00000 56 326.40 0.00000 0.00000 0.00000 57 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 59 326.40 0.00000 0.00000 0.00000 56 326.40 0.00000 0.00000 0.00000 57 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 59 326.40 0.00000 0.00000 0.00000 56 336.40 0.00000 0.00000 0.00000 57 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 59 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 57 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 59 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 0.00000 50 326.40 0.00000 0.00000 0.00000 0.00000 0.000000		39	292.00	0.20004	0.68507	
41 326.40 0.00000 0.00000 42 297.00 0.00000 0.00000 43 298.90 0.00000 0.00000 44 298.90 0.00000 0.00000 45 297.00 0.00000 0.00000 46 297.00 0.00000 0.00000 47 297.00 0.00000 0.00000 48 297.00 0.00000 0.00000 48 297.00 0.00000 0.00000 50 0.00000 50 0.00000 0.00000 50 0.00000 0.00000 50 0.00000 0.00000 50 0.00000 0.00000 51 326.40 0.00000 0.00000 0.00000 53 326.40 0.00000 0.00000 0.00000 55 32 326.40 0.00000 0.00000 55 32 326.40 0.00000 0.00000 55 33 326.40 0.00000 0.00000 55 34 326.40 0.00000 0.00000 55 32 326.40 0.00000 0.00000 56 32 326.40 0.00000 0.00000 57 326.40 0.00000 0.00000 0.00000 58 32 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 59 326.40 0.00000 0.00000 0.00000 0.00000 59 326.40 0.00000 0.00000 0.00000 0.00000 59 326.40 0.00000 0.00000 0.00000 0.00000 59 326.40 0.000000			292.00	0.00000	0.00000	
42 292.00 0.00000 0.00000 43 298.90 0.00000 0.00000 44 298.90 0.00000 0.00000 45 292.00 0.00000 0.00000 46 292.00 0.00000 0.00000 47 292.00 0.00000 0.00000 48 293.50 0.49387 1.6929 49 324.70 0.00000 0.00000 50 292.00 0.00000 0.00000 51 326.40 0.00000 0.00000 52 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 56 326.40 0.00000 0.00000 57 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 59 326.40 0.00000 0.00000 50 326.40 0.00000 0.00000 50 326.40 0.00000 0.00000 50 326.40 0.00000 0.00000 50 326.40 0.00000 0.00000 50 326.40 0.00000 0.00000 50 326.40 0.00000 0.00000 50 326.40 0.00000 0.00000 50 326.50 0.00000 0.00000 50 326.50 0.00000 0.00000 50 326.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.000000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.00000 50 327.50 0.00000 0.000000 50 327.50 0.00000 0.00000 0.00000 50 327.50 0.00000 0.00000 0.000000 50 327.50 0.00000 0.00000 0.00000 50 327.50 0.00000 0.00000 0.00000 50 327.50 0.00000 0.00000 0.00000 50 327.50 0.00000 0.00000 0.00000 50 327.50 0.00000 0.00000 0.00000 50 327.50 0.00000 0.00000 0.00000 50 327.50 0.00000 0.00000 0.000000 50 327.50 0.00000 0.00000 0.000000 0.000000 50 327.50 0.00000 0.000000 0.000000 50 327.50 0.00000 0.000000 0.000000 50 327.50 0.00000 0.000000 0.000000 50 327.50 0.000000 0.000000 0.000000 50 327.50 0.00						
43		41	325.40			
43		42	292.00	. 0.00000	0.00000	
44						
45						
45		44	298.90	0.00000	0.00000	
46					0.00000	
47 292.00 0.00000 0.00000						
48		46	292.00	0.0000		
48		Δ7	292.00	0.00000	0.00000	
\$9						
50 222.00 0.00000 0.00000 51 326.40 0.00000 0.00000 52 336.40 0.00000 0.00000 53 336.40 0.22901 1.00800 54 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 56 326.50 0.00000 0.00000 57 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 58 326.40 0.00000 0.00000 0.00000 59 337.50 0.99805 2.95719 70 326.90 0.00000 0.00000 0.00000 71 347.80 0.00000 0.00000 0.00000 72 336.80 0.00000 0.00000 0.00000 73 326.90 0.00000 0.00000 0.00000 74 347.80 0.00000 0.00000 0.00000 75 326.90 0.00000 0.00000 0.00000 76 326.90 0.00000 0.00000 0.00000 77 326.90 0.00000 0.00000 0.00000 78 326.90 0.00000 0.00000 0.00000 79 326.90 0.00000 0.00000 0.00000 82 32 32.30 0.00000 0.00000 0.00000 83 32 32.30 0.00000 0.00000 0.00000 84 32.30 0.00000 0.00000 0.000000 85 326.30 0.00000 0.00000 0.000000 85 326.30 0.00000 0.00000 0.000000 0.000000 85 326.30 0.00000 0.00000 0.000000 0.000000 85 326.30 0.00000 0.000000 0.000000 0.000000 0.000000						
50 222.00 0.00000 0.00000 51 326.40 0.00000 0.00000 52 326.40 0.00000 0.00000 53 326.40 0.32901 1.00000 54 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 57 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 59 326.40 0.05825 0.18882 60 326.40 0.05825 0.18882 60 326.40 0.05825 0.18882 60 326.40 0.00000 0.00000 61 315.80 0.27672 0.67625 62 326.30 0.00000 0.00000 63 326.40 0.00000 0.00000 64 326.80 0.00000 0.00000 65 326.40 0.00000 0.00000 66 326.40 0.00000 0.00000 67 326.80 0.00000 0.00000 67 326.80 0.00000 0.00000 67 326.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99805 2.95719 70 326.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 326.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 326.90 0.00000 0.00000 77 326.90 0.00000 0.00000 78 326.90 0.00000 0.00000 79 326.90 0.00000 0.00000		49	324.70	0.00000	0.00000	
51 336.40 0.00000 0.00000 52 336.40 0.00000 0.00000 53 336.40 0.00000 0.00000 54 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 56 326.40 0.00000 0.00000 57 336.40 0.00000 0.00000 57 336.40 0.00000 0.00000 58 326.40 0.00000 0.00000 59 326.40 0.0582 0.18882 60 326.40 0.0582 0.18882 61 115.80 0.77672 0.18625 62 360.90 0.00000 0.00000 64 360.90 0.00000 0.00000 66 360.90 0.00000 0.00000 66 360.90 0.00000 0.00000 66 360.90 0.00000 0.00000 67 336.80 0.00000 0.00000 68 326.40 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 341.80 0.00000 0.00000 72 315.80 0.00000 0.00000 73 36.80 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.000000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000					0.00000	
52 336.40 0.00000 0.00000 54 336.40 0.32901 1.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 55 326.40 0.00000 0.00000 57 326.40 0.00000 0.00000 58 326.40 0.00000 0.00000 58 326.40 0.05842 0.3075 58 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.05842 0.3075 59 326.40 0.00000 0.00000 61 315.80 0.27672 0.67625 62 326.30 0.00000 0.00000 63 326.30 0.00000 0.00000 64 326.30 0.00000 0.00000 65 320.50 0.00000 0.00000 66 320.50 0.00000 0.00000 67 326.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99805 2.95719 70 326.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 326.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 326.90 0.00000 0.00000 76 326.90 0.00000 0.00000 77 326.90 0.00000 0.00000 78 326.90 0.00000 0.00000 78 326.90 0.00000 0.00000 79 326.90 0.00000 0.00000 82 32 326.90 0.00000 0.00000 83 326.90 0.00000 0.00000 84 329.30 0.00000 0.00000 85 325.30 0.00000 0.00000 86 325.30 0.00000 0.00000 87 325.30 0.00000 0.00000 88 33 326.90 0.00000 0.00000 89 325.30 0.00000 0.00000 89 325.30 0.00000 0.00000 89 325.30 0.00000 0.00000 89 325.30 0.00000 0.00000 89 325.30 0.00000 0.00000 89 325.30 0.00000 0.00000 99 325.30 0.00000 0.00000 99 325.30 0.00000 0.00000 99 325.30 0.00000 0.00000 99 325.30 0.00000 0.00000 99 325.30 0.00000 0.00000						
52 336.40 0.00000 0.00000		51	326.40	0.00000		
53			326.40	0.00000	0.00000	
\$4 326.40 0.00000 0.00000 \$56 326.40 0.00000 0.00000 \$57 326.40 0.00000 0.00000 \$58 326.40 0.00000 0.00000 \$59 326.40 0.006184 0.15882 \$60 350.90 0.06525 0.18880 \$61 315.80 0.27672 0.37625 \$62 360.90 0.00000 0.00000 \$64 360.90 0.00000 0.00000 \$65 350.50 0.00000 0.00000 \$66 350.50 0.00000 0.00000 \$67 336.80 0.00000 0.00000 \$68 326.40 0.00000 0.00000 \$69 337.50 0.00000 0.00000 \$69 337.50 0.98805 2.95719 \$70 360.90 0.00000 0.00000 \$71 347.80 0.00000 0.00000 \$72 336.80 0.00000 0.00000 \$73 360.90 0.00000 0.000000 \$74 347.80 0.00000 0.00000 \$75 360.90 0.00000 0.00000 \$76 360.90 0.00000 0.00000 \$77 346.90 0.00000 0.00000 \$78 395.50 0.00000 0.00000 \$79 360.90 0.00000 0.00000 \$70 360.90 0.00000 0.00000 \$71 347.80 0.00000 0.00000 \$72 336.90 0.00000 0.00000 \$73 360.90 0.00000 0.00000 \$74 347.80 0.00000 0.00000 \$75 360.90 0.00000 0.00000 \$76 360.90 0.00000 0.00000 \$77 360.90 0.00000 0.00000 \$78 395.50 0.00000 0.00000 \$80 360.90 0.00000 0.00000 \$81 395.50 0.00000 0.00000 \$81 395.50 0.00000 0.00000 \$82 360.90 0.00000 0.000000 \$83 360.90 0.00000 0.00000 \$84 360.90 0.00000 0.00000 \$85 395.50 0.00000 0.00000 \$85 395.50 0.00000 0.00000 \$86 395.50 0.00000 0.00000 \$87 395.50 0.00000 0.00000 \$88 395.50 0.00000 0.00000 \$89 360.90 0.00000 0.00000 \$89 360.90 0.00000 0.00000 \$89 395.50 0.00000 0.00000 \$89 395.50 0.00000 0.00000 \$89 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.000000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.00000 \$80 395.50 0.00000 0.000000 \$80 395.50 0.00000 0.000000 \$80 395.50 0.00000 0.000000 \$80 395.50 0.00000 0.000000 \$80 395.50 0.000000 0.000000 \$80 395.50 0.000000 0.000000 \$80 395.50 0.000000 0.000000 \$80 395.50 0.000000 0.000000 \$80 395.50 0.000000 0.000000 \$80 395.50 0.000000 0.0000000 \$80 395.50 0.000000 0.0000000 \$80 395.50 0.000000 0.000						_
55 326.40 0.00000 0.00000 56 326.40 0.00000 0.00000 57 326.40 0.00000 0.00000 58 326.40 0.065184 0.15882 59 326.40 0.05525 0.18080 61 315.80 0.27672 0.87625 62 360.90 0.00000 0.00000 63 326.40 0.00000 0.00000 64 360.90 0.00000 0.00000 65 326.40 0.00000 0.00000 66 360.90 0.00000 0.00000 67 336.80 0.00000 0.00000 67 336.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 39 360.90 0.00000 0.00000 88 395.30 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000						·
55 326.40 0.00000 0.00000 56 326.40 0.00000 0.00000 57 326.40 0.00000 0.00000 58 326.40 0.065184 0.15882 59 326.40 0.05525 0.18080 61 315.80 0.27672 0.87625 62 360.90 0.00000 0.00000 63 326.40 0.00000 0.00000 64 360.90 0.00000 0.00000 65 326.40 0.00000 0.00000 66 360.90 0.00000 0.00000 67 336.80 0.00000 0.00000 67 336.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 39 360.90 0.00000 0.00000 88 395.30 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000 89 396.90 0.00000 0.00000		54	3 26.40	0.00000	0.00000	•
56 335.40 0.00000 0.00000 58 336.40 0.00000 0.00000 58 336.40 0.09849 0.30175 59 326.40 0.05184 0.15825 60 360.30 0.05525 0.18080 61 315.80 0.27672 0.87625 62 360.90 0.00000 0.00000 63 326.40 0.00000 0.00000 64 360.90 0.49410 1.39698 65 350.50 0.00000 0.00000 66 363.36.80 0.00000 0.00000 67 336.80 0.00000 0.00000 68 336.80 0.00000 0.00000 69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 347.80 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 37 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 396.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000						
57 326.40 0.00000 0.00000 58 326.40 0.09849 0.30175 59 326.40 0.05184 0.15882 60 360.90 0.06525 0.18080 61 315.80 0.27672 0.87625 62 360.90 0.00000 0.00000 64 360.90 0.49410 1.36908 65 350.50 0.00000 0.00000 66 360.90 0.49410 1.36908 66 360.90 0.00000 0.00000 67 335.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99865 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 560.90 0.00000 0.00000 76 369.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 70 360.90 0.00000 0.00000 71 37 360.90 0.00000 0.00000 72 33 360.90 0.00000 0.00000 73 360.90 0.00000 0.00000 74 37 360.90 0.00000 0.00000 75 360.90 0.00000 0.00000 76 369 395.30 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 360.90 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 37 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000						
57 336.40 0.00000 0.00000 58 336.40 0.09499 0.30175 59 336.40 0.05184 0.15882 60 350.30 0.06525 0.18080 61 315.80 0.27672 0.87625 62 360.90 0.00000 0.00000 64 360.90 0.49410 1.36908 65 350.50 0.00000 0.00000 66 360.90 0.49410 1.36908 67 336.80 0.00000 0.00000 68 337.50 0.00000 0.00000 69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 37 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 39 360.90 0.00000 0.00000 88 39 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000		56	326.40			·
58 336, 40 0.09849 0.30175				0.00000	0.00000	
59 326.40 0.05184 0.15882 0.15882 60 360.90 0.06525 0.18080 61 315.80 0.27672 0.87625 0.18080 61 315.80 0.27672 0.87625 62 360.90 0.00000 0.00000 0.00000 64 360.90 0.49410 1.36938 65 350.50 0.00000 0.00000 0.00000 66 360.90 0.00000 0.000						
60 360.90 0.06525 0.18080 61 315.80 0.27677 0.87625 62 360.90 0.00000 0.00000 0.00000 63 3 326.40 0.00000 0.00000 0.00000 0.00000 64 360.90 0.09410 1.36938 65 350.50 0.00000 0.00000 0.00000 0.00000 66 360.90 0.00000 0.00000 0.00000 0.00000 66 336.40 0.00000 0.00						
60 360.90 0.06525 0.18080 61 315.80 0.27677 0.87625 62 360.90 0.00000 0.00000 0.00000 63 3 326.40 0.00000 0.00000 0.00000 0.00000 64 360.90 0.09410 1.36938 65 350.50 0.00000 0.00000 0.00000 0.00000 66 360.90 0.00000 0.00000 0.00000 0.00000 66 336.40 0.00000 0.00		59	326.40	0.05184	0.15882	
61 315.80 0.27672 0.37625 62 360.90 0.00000 0.00000 63 383.40 0.00000 0.00000 64 360.90 0.49410 1.38908 66 360.90 0.00000 0.000000 67 336.80 0.00000 0.00000 68 336.40 0.00000 0.00000 69 337.50 0.98055 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 81 395.30 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 365.90 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.000000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 89 305.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000						
\$22 360.90 0.00000 0.000000 \$64 360.90 0.49410 1.35908 \$65 350.50 0.00000 0.000000 \$66 350.90 0.00000 0.000000 \$67 336.80 0.00000 0.000000 \$68 326.40 0.00000 0.000000 \$69 337.50 0.99805 2.99719 \$70 360.90 0.00000 0.000000 \$71 347.80 0.00000 0.00000 \$72 336.80 0.00000 0.00000 \$73 360.90 0.00000 0.00000 \$73 360.90 0.00000 0.00000 \$74 347.80 0.00000 0.00000 \$75 360.90 0.00000 0.00000 \$75 360.90 0.00000 0.00000 \$75 360.90 0.00000 0.00000 \$76 360.90 0.00000 0.00000 \$77 360.90 0.00000 0.00000 \$78 395.30 0.00000 0.00000 \$80 360.90 0.00000 0.00000 \$80 360.90 0.00000 0.00000 \$81 395.30 0.00000 0.00000 \$82 360.90 0.00000 0.00000 \$83 360.90 0.00000 0.00000 \$84 360.90 0.00000 0.00000 \$85 395.30 0.00000 0.00000 \$85 395.30 0.00000 0.00000 \$88 355.30 0.00000 0.00000 \$89 360.90 0.00000 0.00000 \$80 360.90 0.00000 0.000000 \$80 360.90 0.00000 0.00000 \$80 360.90 0.00000 0.00000 \$80 360.90 0.00000 0.00000 \$80 360.90 0.00000 0.00000 \$80 360.						
62 360.90 0.00000 0.00000 63 336.40 0.00000 0.00000 64 360.90 0.49410 1.36908 65 350.50 0.00000 0.00000 67 336.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 360.90 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000 89 39 395.30 0.00000 0.00000	•	61	315 .80	0.27672	0.87625	
63 326,40 0.00000 0.000000 64 360.90 0.49410 1.35938 66 360.90 0.49410 1.35938 66 360.90 0.00000 0.000000 0.000000 0.000000 0.000000			360.90	0.00000	0.00000	
64 360.90 0.49410 1.36908 65 350.50 0.00000 0.00000 66 360.90 0.00000 0.00000 67 336.80 0.00000 0.00000 68 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 360.90 0.00000 0.00000 88 34 360.90 0.00000 0.00000 88 35 360.90 0.00000 0.00000 88 360.90 0.00000 0.00000 89 37 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000						
65 350.50 0.00000 0.00000 66 360.30 0.00000 0.00000 67 336.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.98805 2.95719 70 360.30 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 74 347.80 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000						
65 350.50 0.00000 0.00000 66 360.30 0.00000 0.00000 67 336.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.98805 2.95719 70 360.30 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 74 347.80 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000 89 396.30 0.00000 0.00000		64	360.90	0.49410	1.36908	
66 360.90 0.00000 0.00000 67 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 79 360.90 0.00000 0.00000 80 350.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 396.90 0.00000 0.00000 87 39 360.90 0.00000 0.00000 88 3 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 80 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 396.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000				0.00000	0.00000	
67 336.80 0.00000 0.00000 68 326.40 0.00000 0.00000 69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 380.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000						
68						
68		67	336.80	0.00000	0.00000	
69 337.50 0.99805 2.95719 70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 38 360.90 0.00000 0.00000 89 38 360.90 0.00000 0.00000 89 38 395.30 0.00000 0.00000 89 380.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 380.90 0.00000 0.00000 89 384.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000						
70 360.90 0.00000 0.00000 71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 80 0.00000 0.00000 81 360.90 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000						÷
71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000		· 69	337.50	0.99805	2.95/19	
71 347.80 0.00000 0.00000 72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000		70	360.90	0.00000	0.0000	
72 336.80 0.00000 0.00000 73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 395.30 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000						
73 360.90 0.00000 0.00000 74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 80 381 360.90 0.00000 0.00000 80 382 360.90 0.00000 0.00000 80 383 360.90 0.00000 0.00000 80 384 360.90 0.00000 0.00000 80 385 395.30 0.00000 0.00000 80 386 360.90 0.00000 0.00000 80 387 395.30 0.00000 0.00000 80 380.90 0.00000 0.00000 80 380.90 0.00000 0.000000 80 380.90 0.00000 0.000000 80 380.90 0.00000 0.000000 80 380.90 0.00000 0.000000 80 380.90 0.00000 0.000000 80 381 380.90 0.000000 0.000000 80 382.20 0.00000 0.000000 0.000000 90 394 394.30 0.000000 0.000000 0.000000 91 382.20 0.000000 0.000000 0.00000000000000						
74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000	•	72	336.80	0.00000	0.00000	
74 347.80 0.00000 0.00000 75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000		73	360.90	0.00000	0.00000	
75 360.90 0.00000 0.00000 76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000						
76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 36 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 80 395.30 0.00000 0.00000 81 395.30 0.00000 0.00000 82 395.30 0.00000 0.00000 83 395.30 0.00000 0.00000 84 395.30 0.00000 0.00000 85 395.30 0.00000 0.00000 86 395.30 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000						
76 360.90 0.00000 0.00000 77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 36 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 80 395.30 0.00000 0.00000 81 395.30 0.00000 0.00000 82 395.30 0.00000 0.00000 83 395.30 0.00000 0.00000 84 395.30 0.00000 0.00000 85 395.30 0.00000 0.00000 86 395.30 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000		75	360.90	0.00000	0.00000	
77 360.90 0.00000 0.00000 78 395.30 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 395.30 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000						·
78 395.30 0.00000 0.00000 79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 36 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000						
79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000		77	360.90	0.00000	0.0000	
79 360.90 0.00000 0.00000 80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000		78	395.30	0.00000	0.00000	
80 360.90 0.00000 0.00000 81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000						
81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
81 395.30 0.00000 0.00000 82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000		80	360.90	0.00000	0.00000	
82 360.90 0.00000 0.00000 83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000						•
83 360.90 0.00000 0.00000 84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000	•	82	360.90	0.00000	0.0000	
84 360.90 0.00000 0.00000 85 395.30 0.00000 0.00000 86 260.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000					0,00000	
85 395.30 0.00000 0.00000 86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000	•					
86 360.90 0.00000 0.00000 87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000		85	395.30	0.00000	0.00000	
87 395.30 0.00000 0.00000 88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						·
88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
88 395.30 0.00000 0.00000 89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000		87	395.30			
89 360.90 0.00000 0.00000 90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000				0-00000	0.00000	
90 395.30 0.00000 0.00000 91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						•
91 360.90 0.00000 0.00000 92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000		90	395.30	0.00000	0.00000	
92 394.30 0.00000 0.00000 93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
93 394.30 0.00000 0.00000 94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
94 394.30 0.00000 0.00000 95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000			394.30	0.00000	0.00000	
95 382.20 0.00000 0.00000 96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000						
96 429.80 0.00000 0.00000 97 360.90 0.00000 0.00000		95	382.20			
360.90 0.00000 0.00000				0.00000	0.00000	
98 395.30 0.00000 0.00000						
		98	` 395.30	0.00000	,0.0000	
					•	

100	395.30	0.00000	0.00000
101	429.80	0.00000	0.00000
102	395.30	0.00000	0.00000
103	395.30	0.00000	0.00000
104	395.30	0.00000	0.00000
105	429.80	0.00000	0.00000
106	395.30	0.00000	. 0.00000
107	395.30	0.00000	0.00000
108	429.80	0.00000	0.00000
109	429.80	0.00000	0.00000
110	429.80	0.00000	0.00000
111	395.30	0.00000	0.00000
112	429.80	0.00000	0.00000
113	464.20	0.00000	0.00000
114	464.20	0.00000	0.00000
115	429.80	0.00000	0.00000
116	429.80	0.00000	0.00000
117	464.20	0.00000	0.00000
118	498.60	0.00000	0.00000

CONCENTRATION = 3.657 NG/L

TOTAL PICOMOLES = 0.0117

AVERAGE MOLECULAR WEIGHT = 313.8

NUMBER OF CALIBRATED PEAKS FOUND= 12

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET SCHENECTADY, NY 12305 (518) 346-4592

CONGENER WEIGHT and MOLE REPORT

NEA FILE NAME: 0515BW1.mol

CUSTOMER:

CIBA-GIEGY CORPORATION

SAMPLE DESCRIPTION: 920515BW1E W/B

COMMENT:

CRANSTON WATER COLUMN PROJECT PCB H2O SAMPLES COC:5/14/92

DATE ACQUIRED:

19-MAY-1992 5:24

TYPE FOR MIXED PEAK DECONVOLUTION= S

PEAK#	RET. TIME	T-CL:0-CL	TUPAC#	RRT	CONGENERS	WEIGHT %	MOLE %	PEAK#
<u> </u>	0.00	0:0	000	.0997	BIPHENYL	0.000	0.000	1
2	0.00	1:1	001	.1544	2	0.000	0.000	2
3	0.00	1:0	002	.1937	3	0.000	0.000	3
4	0.00	1:0	003	.1975	4	0.000	0.000	4
5	0.00	2:2	004 010	. 2245	22' ; 26	0.000	0.000	5
6	0.00	2:1	007 009	. 2566	24 ; 25	0.000	0.000	6
7	0.00	2:1	006	.2709	23'	0.000	0.000	7
8	0.00	2:1	005 008	.2785	23 ; 24'	0.000	0.000	8
9.	0.00	2:0	014	.2973	35	0.000	0.000	9
10	0.00	3:3	019	. 3045	22'6	0.000	0.000	10
11	0.00	3:2	030	.3165	246	0.000	0.000	11
12	0.00	2:0	011	.3238	33'	0.000	0.000	12
13	0.00	2:0	012 013	.3297	34 ; 34'	0.000	0.000	13
14	0.00	3:2 2:0	018 015	.3387	22'5 ; 44'	0.000	0.000	14
15	0.00	3:2	017	.3398	22'4	0.000	0.000	15
16	0.00	3:2	024 027	3508	236 ; 23'6	0.000	0.000	16
17	0.00	· 3 :2	016 032	. 3625	22'3 ; 24'6	0.000	0.000	1.7
18	0.00	3:1	023	. 3770	. 235	0.000	0.000	18
19	0.00	3:1 4:4	034 054	.3800	2'35 ; 22'66'	0.000	0.000	19
20	0.00	3:1	029	.3820	245	0.000	0.000	20
21	0.00	3:1	026	.3911	23'5	0.000	0.000	21
22	0.00	3:1	025	.3937	23'4	0.000	0.000	22
23	23.89	3:1	031	.4024	24'5	7.384	9.000	23
24	23.94	3:1 4:3	028 050	. 4031	244'; 22'46	5.0 29	6.129	24
25	0.00	3:1.4:3	021 033	.4170	233' ; 234 ; 22'56'	0.000	0.000	25
26	0.00	3:1 4:3	022 051	.4267	234' ; 22'46'	0.000	0.000	26
27	0.00	4:3	045	.4334	22'36	0.000	0.000	27
28	0.00	3:1	036	4379	33*5	0.000	0.000	28
29	0.00	4:3	046	. 4450	22'36'	0.000	0.000	29
30	0.00	3:1	039	. 4488	34'5	0.000	0.000	30
31	0.00	4:2	052 073	. 4554	22'55' ; 23'5'6	0.000	0.000	31
32	0.00	4:2	049	.4610	22145	0.000	0.000	32

34	0.00	4:2	048 075	.4651	22'45 ; 244'6 2346 ; 2356 33'4 22'466' ; 22'35' 344' ; 22'34' ; 233'6	0.000	0.000	34
35	0.00	4:2	065 062	. 4865	2346 : 2356	0.000	0.000	35
36	0.00	3:0	035	.4738	33'4	0.000	0.000	,36
37	25.76	5:4 4:2	104 044	.4832	22'466' : 22'35'	5.361	5.762	37
38	0.00	3:0 4:2	037 042	. 4870	344' : 22'34' : 233'6	0.000	0.000	38
39	26.16	4:2	064 071	.4990	22174 - 2246 - 22146 - 2	E AEC	5.878	39
40	0.00	4:1	068	.5040	23'45' ? 22'366' 22'33' 22'45'6; 233'5 22'44'6; 23'4'5 233'5'; 234'5 244'5; 22'356' 23'4'5; 2'345; 2345? 23'44'; 22'356; 22'35'6 22'34'6; 22'3'46; 233'4	0.000	0.000	40
41	0.00	5:4		.5057	22'366'	0.000	0.000	41
42	0.00	4:2		.5102	22:33:	0.000	0.000	42
43	0.00	5:3 4:1	103 057	.5155	22 33	0.000	0.000	
44	0.00	5:3 4:1	100 067	.5212	22 43 0 7 233 3	0.000		43
45	0.00	4:1	058 063	.5212	24 11 0 , 43 4 3	0.000	0.000	44
46	0.00	4:1 5:3	074 094	.5340	233 3 ; 234 3	0.000	0.000	45
47	0.00	4:1 3:3 4:1	070 061		244 0 ; 44 300	0.000	0.000	46
48	27.23	4:1 4:1 5:3		.5407	23 4 3 ; 4 343 ; 2343?	0.000	0.000	47
	0.00	5:3 4:1	066 095	.5447	23,44 ; 24,330 ; 22,330	13.503	14.438	48
49 50	0.00		091 098	.5549	22'34'6 ; 22'3'46 ; 233'4 233'4' ; 2344'	0.000	0.000	49
		4:1	056 060	.5676	23.4 ; 234	0.000	0.000	50
51	0.00	6:4 5:3	155 084	.5666	22'44'66'; 22'33'6; 22'355' 22'346' 22'34'5; 22'455' 22'44'5	0.000	0.000	51
52	0.00	5:3	089	.5779	42° 346°	0.000	0.000	52
53	28.06	5:2		.5814	22'34'5 ; 22'455'	8.996	8.649	53
54	0.00	5:2	099	.5880	22'44'5	0.000	0.000	54
55	0.00	6:4 5:2	150 112	.5969	0 44 CA . OL CLA . OU PC AA	17.1887	0.000	55
56	0.00	5:2	083 109	.6029	22'33'5 ; 233'46	0.000	0.000	56
57	0.00	6:4 5:2	152 097	.6062	22'33'5; 233'46 22'3566'; 22'345; 22'3'45	0.000	0.000	57
58	28.82	5:2	087 111	.6175	22'345'; 233'55'; 2344'6 22'344'; 23456? 22'33'66' 33'44'; 233'4'6 22'44'56' 22'33'4 22'355'6	2.693	2.589	58
59	28.97	5:2	085 116	.6224	22'344' ; 23456?	1.417	1.363	59
60	29.06	6:4	136	.6257	22'33'66'	1.784	1.551	60
61	29.16	4:0 5:2		.6295	33'44' ; 233'4'6	7.566	7.519	61
62	0.00	6:3	154	.6349	22'44'56'	0.000	0.000	62
63	0.00	5:2	082	.6453	22'33'4	0.000	0.000	63
64	29.74	6:3	151	.6499	22'355'6	13.509	11.747	64
65	0.00	6:3 5:1	135 124	.6563	44 33 30 ; 4 344 3	0.000	0.000	65
66	0.00	6:3	144	.6584			. 0.000	66
67	0.00	5:1 6:3	107 108	.6628	233'4'5 ; 233'45' ; 22'34'56	0.000	0.000	67
68	0.00	5:1	123	.6658	2'344'5	0.000	0.000	68
69	30.16		149 118	.6672	22'34'5'6 ; 23'44'5 ; 233'45	27.288	25.374	69
70	0.00	6:3	139 140		22'344'6 ; 22'344'6'	0.000	0.000	70
71	0.00	6:3 5:1	134 143	.6796	22'33'56' ; 22'3456'; 2344'5		0.000	<i>7</i> l
72		5:1 6:3	122 131	.6871	2'33'45; 22'33'46;22'33'55'+		0.000	72
73	0.00	6:2	146 161	.6955	22'34'55' ; 233'45'6	0.000	0.000	73
74	0.00	6:3 5:1	132 105	.7035	22'33'46' ; 233'44'	0.000	0.000	74
75	0.00	6:2	153	.7036	22'44'55'	0.000	0.000	75
76	0.00	6:2	168	.7068	23'44'5'6	0.000	0.000	76
77	0.00	6:2	141	.7203	22'3455'	0.000	0.000	77
78	0.00	7:4	179	.7205	22'33'566'	0.000	0.000	78
79	0.00	6:2	130	.7284	22'33'45'	0.000	0.000	79
80	0.00	6:2	137	.7329	22'344'5	0.000	0.000	80
81	0.00	7:4	176	.7305	22' 33 '466'	0.000	0.000	81
82	0.00	6:2	138 163	.7403	22'344'5' ; 233'4'56 ; +2	0.000	0.000	82
83	0.00	6:2	158	.7429	233'44'6	0.000	0.000	83
84	0.00	6:2	129	.7501	22'33'45	0.000	0.000	84
85	0.00	7:3	. 178	.7537	22'33'55'6	0.000	0.000	85
86	0.00	6:2	166	.7572	2344'56	0.000	0.000	86
87	. 0.00	7:3	- 175	.7611	23'44'56 22'33'45'6 22'34'55'6 ; 22'344'56'	0.000	0.000	87
88	0.00	7:3	187 182	.7653	22'34'55'6 ; 22'344'56'	0.000	0.000	88
89	0.00	6:2	128	.7761	22'33'44'	0.000	0.000	89
90	0.00	7:3	183	.7720	22'344'5'6	0.000	0.000	90
91	0.00	6:1	167	.7814	23'44'55'	0.000	0.000	91
92	0.00	7:3	185	.7848	22'3455'6	0.000	0.000	92
93	0.00	7:3	174 181	.7965	22'33'456' ; 22'344'56	0.000	0.000	93
94	0.00	7:3	177	.8031	22'33'4'56	0.000	0.000	94
95	0.00	7:3 6:1	171 156	.8105	22'33'44'6 ; 233'44'5	0.000	0.000	95
96	0.00	8:4	202	.8089	22'33'55'66'	0.000	0.000	96
97	0.00	6:1	157	.8184	233!44'5'	0.000	0.000	97
98	0.00	7:3	173	.8152	22'33'456	0.000	0.000	98
								-

1

-

100	0.00	7:2	172 192	.8278	22'33'455' ; 233'455'6	0.000	0.000	100
101	0.00	8:4	197	.8293	22'33'44'66'	0.000	0.000	101
102	0.00	7:2	180	.8362	22'344'55'	0.000	0.000	102
103	0.00	7:2	193	.8397	233'4'55'6	0.000	0.000	103
104	0.00	7:2	191	.8447	233'44'5'6	0.000	0.000	104
105	0.00	8:4	199	. 8494	22, 33, 4566	0.000	0.000	105
106	0.00	7:2	170	.8740	22'33'44'5	0.000	0.000	106
107	0.00	7:2	190	.8740	233'44'56	0.000	0.000	107
108	0.00	8:3	198	. 8845	22'33'455'6	0.000	0.000	108
109	0.00	8:3	201	.8875	22'33'4'55'6	0.000	0.000	109
110	0.00	8:3	196 203	.8935	22'33'44'5'6 ; 22'344'55'6	0.000	0.000	110
111	0.00	7:1	189	.9142	233'44'55'	0.000	0.000	111
112	0.00	8:3	195	.9321	22'33'44'56	0.000	0.000	112
113	0.00	9:4	208	.9320	22'33'455'66'	0.000	0.000	113
114	0.00	9:4	207	.9423	22'33'44 '56 6'	0.000	0.000	114
115	0.00	8:2	194	.9620	22'33'44'55'	0.000	0.000	115
116	0.00	8:2	205	.9678	233'44'55'6	0.000	0.000	116
117	0.00	9:3	206	1.010	22'33'44'55'6	0.000	0.000	117
118	0.00	10:4	209	1.050	22'33'44'55'66'	0.000	0.000	118

CONCENTRATION = 3.657 NG/L

TOTAL PLOOMOLES = 0.0117

AVERAGE MOLECULAR WEIGHT = 313.8

NUMBER OF CALIBRATED PEAKS FOUND= 12

NORTHEAST ANALYTICAL, INC.

301 NOTT STREET SCHENECTADY, NY 12305 (518) 346-4592

PCB SUMMARY REPORT

NEA FILE NAME: 0515BWl.hrs

CUSTOMER:

CIBA-GIEGY CORPORATION

SAMPLE DESCRIPTION: 920515BW1E W/B

COMMENT:

CRANSTON WATER COLUMN PROJECT PCB H2O SAMPLES COC:5/14/92

DATE ACQUIRED:

19-MAY-1992 5:24

Total PCBs in Sample= 3.66 NG/L

PCB Homolog Distribution

NOMINAL 'AROCLOR' Distribution

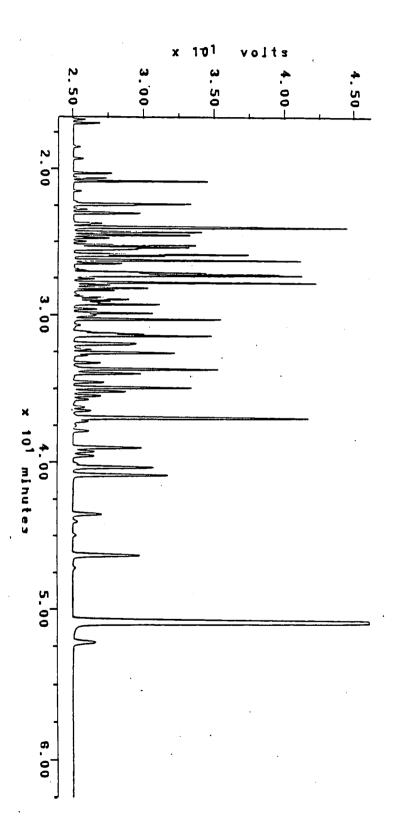
Homologs	Wt. % N	Mole %	Aroclor	Indicator Peak	Amount	Perc	ent
	· ·			(PK#/IUPAC#)	NG/L	SEDIMENT	ВІОТА
Mono	0.00	0.00	A1221	2/001	0.0000	0.0	0.0
Di	0.00	0.00	A1242	23+24/31+28	0.4540	62.1	31.3
Tri	12.41	15.13	Al254SED		0.2767	37.9	
Tetra	23.73	25.43	A1254BIO	***/###	0.9980		68.7
Penta	39.84	38.02	A1260	102/180	0.0000	0.0	0.0
Hexa	24.03	21.42	A1268	115/194	0.0000	0.0	0.0
Hepta	0.00	0.00	11				
Octa	0.00	0.00	11				
Nona	0.00	0.00					
Deca	0.00	0.00					
			***/#	##:69+75+82/149	+153+138		

Ortho Cl / biphenyl Residue = 1.77

Meta + Para Cl / biphenyl Residue = 2.88

TOTAL Cl / biphenyl Residue = 4.66

Sample: PER CHK 1.22PPM Channel: detector 1 Filename: PC042TA
Acquired: 28-APR-82 4:23 Method: Di\MAX\GC1\OGW042TA Operator: NEW
Comments: NORTHEAST ANALYTICAL 301 NOTT STREET SCHENECTADY, NY 518-348-4582



LABORATORY PERFORMANCE AND AUDITS

LABORATORY PERFORMANCE AND AUDITS

Internally, lab performance is monitored by measurement of quality control samples and review of quality control data and calculations. Laboratory method blanks are extracted with each extraction batch to monitor and determine if contamination exists. Duplicate samples are extracted at a frequency of 5% to measure homogeneity of sampling procedures and precision of methodology and analysis. Matrix spike samples are extracted at a refquency of 10% to track recovery and measure method applicability to the matrix accuracy.

The laboratory is certified by the New York State Department of Health which administers the Environmental Laboratory Approval Program (ELAP). Yearly the laboratory is inspected by an ELAP representative who reviews current analyte certifications and inspects log books, record books, and quality control documentation. On a quarterly basis the lab is sent performance evaluation (PE) samples. Samples are analyzed and results reported to ELAP. Certification is maintained by receiving passing scores for submitted results. Our certifications and PE results are on file for client review.

INSTRUMENT MAINTENANCE

INSTRUMENT MAINTENANCE

The laboratory staff is familiar with the analytical instrumentation and required maintenance. The lab has full maintenance contracts from the instrument vendors to insure the minimum amount of instrument down time. In addition sample flow through the laboratory is kept at a high level by having instrument redundancy. A complete inventory of spare parts and consumables is stocked for each instrument. The following instrument maintenance guidelines have been adopted.

INSTRUMENT MAINTENANCE SCHEDULE

INSTRUMENT MAINTENANCE SCHEDULE

Area .	Procedure	Interval
Instruments	alavaina	as required
external surface	cleaning	as required
GC terminal		
paper	replacement	as required
printhead	replacement	as required
GC Filters		
moisture trap	replacement	6 months
carbon trap	replacement	6 months
oxygen trap	replacement	6 months
carrier gas	leak check	as required
GC column	conditioning	noisy baseline/column change
GC column	replacement	loss of performance
Injection Port		•
septum	replacement	retention time shifts
		or every 50 injections
Autosampler		•
syringe	positioning	at installation
syringe	replacement	if damaged or leaking
Detector		ji k
ECD	leak check	at column installation
ECD	thermal clean	1 month
ECD	NRC wipe test	6 months

LABORATORY FACILITIES, INSTRUMENTATION, AND PERSONNEL

LABORATORY FACILITIES, INSTRUMENTATION, AND PERSONNEL

FACILITIES:

Northeast Analytical Inc. has a modern 6000 sq. ft. laboratory located in the city of Schenectady, NY. The Lab is centrally located and in close proximity to the cities of Albany and Troy, and 3.5 hours North of New York City by car. Amtrack is a four block walk and the Albany County Airport is 20 minutes by car.

Currently the laboratory specializes in high resolution, trace PCB analysis on soil, sediment, water, biota and oil. The main laboratory occupies 1100 sq. ft. with a separated data control room for data acquisition and management. Quality control office, main reception area and stock room are also located on the third floor level.

The second floor level houses Northeast Analytical's Trace Level Water laboratory, along with our Metals laboratory. Other facilities include a 10° x 14° walk-in cooler, 12° x 14° walk-in freezer, sample login and storage area, waste storage area, and ultra pure 18 meg water system.

Northeast Analytical is presently under construction to house a new GC/MS and Volatile Organics lab, which will contain a air handling system to assist in trace level analysis.

CERTIFICATION:

Northeast Analytical, Inc. is certified by the State of New York Department of Health under the Environmental Laboratory Approval Program.

INSTRUMENTATION:

Varian 3400 Gas Chromatograph equipped with Varian 8035 Autosampler (60 position), two Ni-63 electron capture detectors, SPI injector, and Universal injector.

Varian 3400 Gas Chromatograph equipped with Varian 8035 Autosampler (60 position), Ni-63 electron capture detector, FID detector, SPI Injector, and Megabore injector:

Varian 3400 Gas Chromatograph equipped with PID detector, HECD detector, cryogenic controlled oven, and two universal injectors.

Tekmar LSC 2000 Purge and Trap Unit

Tekmar ALS 2016, 16 stage Purge and Trap Autosampler.

Tekmar 1000M Capillary Cryogenic Interface for capillary column purge and trap.

Varian 3400 Gas Chromatograph equipped with Varian 8035 Autosampler (60 position), two Ni-63 electron capture detectors, split/splitless injector, and universal injector.

Varian 3400 Gas Chromatograph equipped with 8100 Autosampler (48 position, low level), with Ni-63 electron capture detector and FID detector, split/splitters injection and universal injector.

Zymark Turbovap (3 units), 6-position solvent evaporation device.

Varian SpectrAA-400 Flame Atomic Absorption Spectrometer equipped with Austin 286 personnel computer, Varian PSC-56 programmable sample changer (66 position), and Varian VGA-76 cold vapor generator accessory.

Varian SpectrAA-400 Graphite Furnace Atomic Absorption Spectrometer, equipped with IBM 286 personnel computer, Zeeman background correction, Super lamp accessory, and Varian 50 position autosampler.

Varian Saturn II GC/MC equipped with Varian 3400 GC, Compaq 386-20 computer, Tekmar LSC-2000 and ALS-2016 Purge and Trap Autosampler, Libraries include (NIST, TX, GP and TR).

Thermolyne Model 30400 Furnace.

Dynamic Solutions Maxima 820 Workstation for gas chromatograph data management, equipped with NEC 286 personnel computer:

Dynamic Solutions Maxima 820 Workstation for gas chromatograph data management, equipped with Austin 386 personnel computer.

Novel Network accessing the following computers: 2-ACMA 486-33, 2-Bitwise 386-33, 1-Austin 386-25, 2-Bitwise 386-25, 1-Austin 386-20, 1-NEC-286 and 1-Austin 286 computer.

Bruning Model 912 High Speed X-Y Plotter

Fisher Model 200 Oven

VWR Model 1370GD Oven Mettler PJ3600 DeltaRange Balance

A and D Model ER-180A Balance

Other equipment:

VWR 1370F0 OVEN

OKIDATA OL820

PERSONNEL:

Northeast Analytical's major function is to provide high quality analytical testing services. To meet and maintain quality services, Northeast Analytical employs advanced degreed scientists well versed in environmental and analytical chemistry. Experience of the Senior Chemists plays an important role in project supervision and training Lab Technicians to perform chemical analyses correctly and produce highly respected analytical information.

Resumes for Northeast Analytical's staff are presented on the following pages.

ROBERT E. WAGNER: LABORATORY DIRECTOR

PROFESSIONAL EXPERIENCE:

1989-Present:

Lab Director

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

President and Lab Director of Northeast Analytical, Inc.; supervises and manages laboratory staff in the chemical analysis of various environmental samples for PCB, pesticides, and volatiles; develops and implements analytical methods and procedures; training of laboratory analysts; project manager responsibilities; responsible for review of all data and QA/QC programs; long range planning; client maintenance activities; equipment evaluation and recommendation; maintain laboratory certification programs.

<u> 1981-1989:</u>

Associate Staff Analytical Chemist

General Electric Company
Research & Development Center
P.O. Box 8; 1 River Road
Schenectady, New York 12301

Employed in the field of environmental chemistry (PCB, PCDF, DIOXIN) and analytical polymer chemistry (trace analysis, residue solvent characterization, toxic component analysis). Involved with development of multi-component analysis and synthetic chemistry of standards. Isolation and fractionation of environmental residues from complex matrices. Development of state of the art analytical techniques with high resolution capillary gas chromatography utilizing a variety of detectors (ECD, FID, TCD, HECD). Knowledge of GC and HPLC (H/P, Varian, Shimadzu) and integration systems. In-depth maintenance capability to board level and hands-on hardware configuration.

1978-1981:

Biochemistry Project Supervisor
Albany Medical College of Union University

Albany, New York 12208

Coordinate and develop a federally funded research project investigating the aging process using hamsters as models. Companion study exploring the effects of lipid enriched diets on the lifespan of these animals. Supervisor of three technicians in the daily work routine. Also conducted clinical research to develop a quick and accurate determination of serum bilirubin levels based on a fluorescence quenching method.

1975-1978:

Biochemistry Technician

Albany Medical College of Union University

Albany, New York 12208

Technician on above mentioned federal research project. Preparation of enzyme complexes from various organ tissue for extensive biochemical analysis. Experience with centrifuges, A.A., Spectrophotometers, and fluorometer.

EDUCATION:

1975:

B.S. Chemistry
SUNY at Pottsdam
Pottsdam, New York

PROFESSIONAL AFFILIATIONS:

American Chemical Society Schenectady Chamber of Commerce

PUBLICATIONS:

1984

- 1. J.F. Brown Jr., R.E. Wagner, D.L. Bedard, M.J. Brennan, J.C. Carnahan, R.J. May, and T.J. Tofflemire, "PCB Transformations in Upper Hudson Sediments, Northeast Environmental Science, Vol. 3, No. 384, p.167-179, 1984.
- 2. R.E. Wagner and J.C. Carnahan, "Capillary GC-Quantitation of Polychlorinated Biphenyls, Presented before the Varian Chromatography Symposium, April 1984.

1985

- 2. B.J. Vivier and R.E. Wagner, "Gas Chromatography Data Reduction Software", GE CR&D Memo Report #MOR-85-024, Class 2, March 1985.
- 4. J.F. Brown Jr., R.E. Wagner, D.L. Bedard, M.J. Brennan, J.C. Carnahan, R.J. May, and T.J. Tofflemire, "PCB Dechlorination in Upper Hudson Sediments", Presented before the Division of Environmental Chemistry, American Chemical Society 189th National Meeting, Miami, Florida, Vol. 25, No. 1, Paper #12, p.35-37, April 29-May 3, 1985.
- 5. R.E. Wagner, J.C. Carnahan and R.J. May, "Calibration Method for PCB Analysis by Capillary GC and Electron Capture Detection", Presented before the Division of Analytical Chemistry, American Chemical Society 189th National Meeting, Miami, Florida, Paper #201, April 28-May 3, 1985.
- 7. R.E. Wagner and J.C. Carnahan, "An Internal Standard for Capillary GC Analysis of PCB Mixtures", Presented before the Division of Analytical Chemistry, American Chemical Society 189th National Meeting, Miami, Florida, Paper #202, April 28-May 3, 1985.
- 8. D.L. Bedard, L.H. Bopp, M.J. Brennan, R.E. Brooks, J.F. Brown Jr., J.C. Carnhan, D.T. Gibson, M.L. Haberl, C. Johnson, S.H. Lockwood, K.L. Longley, R.J. May, D.P. Mobley, L.M. Nadium, M.J. Schocker, T. Su, R. Unterman, R.E. Wagner, and G.W. Yeager, General Electric Company Research and Development Program for the Destruction of PCBs, Fourth Progress Report, 6/1/84-6/1/85.
- 9. R.E. Wagner and J.C. Carnahan, "An Internal Standard for Capillary GC Analysis of PCB Mixtures", Presented before the General Electric Company-Wide Materials Characterization Symposium, 7th Meeting, April 22-24, 1985.
- 10. R.E. Wagner and J.C. Carnahan, "Internal Standard for Capillary GC Analysis of PCB Mixtures", Presented before the Varian Chromatography Symposium, March 1985.
- 11. J.F. Brown Jr., D.L. Bedard, L.H. Bopp, J.C. Carnahan, R.W. Lawton, R.D. Unterman, and R.E. Wagner, "Human and Environmental Biodegradation of PCBs", Presented before the EPRI PCB Conference, Seattle, Washington, October 22, 1985.
- 12. J.F. Brown Jr., D.L. Bedard, J.C. Carnahan, R.W. Lawton, R.E. Wagner, "Environmental Dechlorination of PCBs", Presented before the Society for Environmental Toxicology and Chemistry Meeting, St. Louis, Missouri, November 11, 1985

13. J.F. Brown Jr., R.E. Wagner, "Polychlorinated Biphenyl (PCB) Movement and Transformation in Acushnet Estuary Sediments", 9/26/86. A draft manuscript prepared for RTE Aerovox Corporation and GE/CEIP to be eventually written into two or more scientific paper discripting the above title.

1987

- 14. D.L. Bedard, M.J. Brennan, R.E. Wagner, M.L. Haberl, and J.F. Brown Jr., "Extensive Degradation of Aroclors and Environmentally Transformed PCBs by ALCALIGENES EUTROPHUS H850", Applied and Environmental Microbiology, Vol. 53(5), p.1103-1112, May 1987.
- 15. J.F. Brown Jr., R.E. Wagner, H. Feng, D.L. Bedard, M.J. Brennan, J.C. Carnahan, and R.J. May, "Environmental Dechlorination of PCBs", Environmental Toxicology and Chemistry, Vol. 6(8), p.579-593, August 1989.
- 16. J.F. Brown Jr., D.L. Bedard, M.J. Brennan, J.C. Carnahan, H. Feng, R.E. Wagner, "Polychlorinated Biphenyl Dechlorination in Aquatic Sediments", Science, Vol. 236, p.709-712, May 1987.
- 17. J.F. Brown Jr., J.C. Carnahan, S.B. Dorn, J.T. Groves, W.V. Ligon, Jr., R.J. May, R.E. Wagner, and S.B. Hamilton, "The Levels of Toxic PCDF Congeners in Used and Unused PCB Dielectric Fluids", Dioxin '87 Seventh International Symposium, Las Vegas, Nevada, Oct. 4-9, 1987.
- 18. J.F. Brown Jr., and R.E. Wagner, "PCB Dechlorination in Marine Sediments", Abstracts of Papers at the 8th Annual Meeting of the Society for Environmental Toxicology and Chemistry, Pensacola, FL, No. 136, 1987.

<u> 1988</u>

- 19. J.F. Brown Jr., R.W. Lawton, M.R. Ross, J. Feingold, R.E. Wagner, and S.B. Hamilton, "The Persistence of PCB Congeners in Normal and PCDF-Poisoned Humans", Presented before the DIOXIN '88 Conference, Umea, Sweden, August 21-26, 1988.
- 20. J.F. Brown Jr., and R.E. Wagner, "A Simple Index for Summarizing the Alteration State of Environmental PCB Mixtures", Presented before the Society for Environmental Toxicology and Chemistry Meeting, November 1988.
- 21. J.F. Brown Jr., J.C. Carnahan, S.B. Dorn, J.T. Groves, M.V. Ligon, R.J. May, R.E. Wagner, and S.B. Hamilton, "Levels of Bioactive PCDF Congeners in PCB Dielectric Fluids from Capacitors and Transformers", Chemoshere.
- 22. J.F. Brown Jr., R.E. Wagner, and D.L. Bedard, "PCB Dechlorination in Hudson River Sediment", Science, Vol.240, 1988, p.1674-1676.

<u> 1990</u>

23. J.F. Brown Jr., and R.E. Wagner, "PCB Movement, Dechlorination, and Detoxication in the Acushnet Estuary", Environ. Tox. and Chem., Vol.9, p.1215-1233, 1990.

ROBERT W. STOLL: FINANCIAL MANAGER

PROFESSIONAL EXPERIENCE:

1989-Present:

Financial Manager

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Vice-President and Financial Manager of Northeast Analytical, Inc. Supervises and manages administrative personnel; performs all ordering and purchasing; accounting and business management responsibilities; interface with day to day lab activities; long range planning; client maintenance activities; equipment and annual budget regulation; staffing; and acts as project manager on laboratory projects.

1985-1989:

Process Engineer

General Electric Company

Product Operations Department (POD)

Research and Development Center

P.O. Box 8; 1 River Road Schenectady, New York 12301

Responsible for the operation of the Thin Film Deposition Area of the liquid Crystal Display Pilot Line. Characterization and process control on all films used in the fabrication of Thin Film Field Effect Transistors using Sputtering, Evaporation, and PECVD. Responsible for the daily work routine of four technicians, as well as the equipment, facilities and operator specification for the US factory design.

1980-1985:

Associate Staff Chemist
General Electric Company
Research and Development Center
P.O. Box 8; 1 River Road
Schenectady, New York 12301

CVD Tungsten Project: Developed a unique CVD reactor that was capable of selectively depositing tungsten in 2 micron, straight side wall vias; at deposition rates in excess of 1000A/min. The technology was sold to GENUS Corp. Developed both selective and non-selective tungsten processes using surface pretreatments to either enhance or hinder deposition. Analyzed and developed a method of relieving the tungsten film stress which was responsible for the spontaneous delamination of tungsten films on glass surfaces.

Fumed Silica Project: Designed a high temperature infrared flow cell (750C) and a fluidized bed reactor to study the kinetics of fumed silica using a variety of silylating agents. Analyses were conducted using an Nicolet 7199 FT-IR spectrometer and a Micromeritics Digisorb 2500 Surface Area Analyzer. Responsible of the operation and maintenance of both machines.

Translex Project: Synthesis of polycarbonate via the melt polymerization method. The polymers were analyzed for impurities and thermal degradation products utilizing GC, HPLC, and NMR.

1979-1980:

Research Technician
State University of New York at Albany

1400 Washington Avenue Albany, New York 12222

Provided technical support for a federally funded research project investigating the aging process using hamsters as models. Companion study exploring the effects of lipid enriched diets on the lifespan of these animals. Preparation or enzyme complexes from the heart organ for extensive biochemical analysis. Experience with centrifuges, A.A., spectrophotometers, fluorometers, and oxygraphs.

1976-1977:

Chemical Laboratory Technician

Corning Glass Works

Sullivan Park Research and Development

Painted Post, New York 14870

Responsible for the precision hydrofluoric acid etching of Vycor glass tubes. Operated two computerized lathes used to place layers of BC13, GeC14, and PCL5 on the inside of the Vycor tubes, which were later drawn into optical waveguide fibers.

1972-1975:

Inventory Management Specialist

Military Service

United States Air Force Rank: Sergeant E-4

Non-commissioned officer in charge of the Base Composite Tool Kit Program. Duties involved meeting with representatives of the base maintenance organizations to determine their tool requirements. Maintaining and updating all records for each composite tool kit. Supervised two airman in their daily routine.

EDUCATION:

<u> 1979:</u>

B.S. Chemistry SUNY at Albany Albany, New York

PUBLICATIONS:

Coauthor of 1 journal article and 4 published proceedings of meetings.

PATENTS:

Method for Treating Fumed Silica (U.S.Pat.4,554,147)

Enhancing the Selectivity of Tungsten Deposition on Conductor and Semi-conductor Surfaces (U.S.Pat.4,552,783)

Selective Chemical Vapor Deposition Apparatus (U.S.Pat.4,653,428)

Method for Selective Deposition of Tungsten by Chemical Vapor Deposition onto Metal and Semi-conductor Surfaces (U.S.Pat.4,471,928)

GREGORY A. YOGIS: LABORATORY MANAGER

PROFESSIONAL EXPERIENCE:

1991-PRESENT:

Organics Laboratory Manager Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Manages all personnel and sample flow of the organics laboratory.

1987-1991:

Supervisor, Gas Chromatography/Senior Chemist

ABB Environmental Services, Inc.

Portland, Maine

Supervisor of analytical section: Training, quality assurance/control, scheduling, and review. Analytical methods: Extensive method development, analysis and troubleshooting utilizing 500, 600, and SW-846, USEPA CLP and NYS-DEC CLP series methods. Developed and conducted soil gas field methods and other screening programs. Coordination of laboratory information systems: Setup and support of laboratory computer software and hardware. Including implementation, coordination and troubleshooting of USEPA/CLP GC and GC/MS computer format deliverables. GC/MS analysis: Analysis using GC/MS VOA methods 624 and 8240. Laboratory Safety Officer: Developed and coordinated lab health and safety programs.

1984-1987:

Research Associate

State University of New York Research Center at Oswego

Oswego, New York

Pesticide/PCB Analytical Method Development: Biogeochemical uptake studies of organochlorine contaminants. Extensive GC and extraction method development. Laboratory Supervision: Assisted in laboratory management and supervised and instructed technical assistants. Remote sensing: Extensive bioacoustic method development and field work, USEPA water chemistry and computer data programming and analysis.

1983-1986:

Thesis Research/Teaching Assistant

Chemistry Department

State University of New York at Oswego

Oswego, New York

Thesis research: Kinetic studies of Cobolt III complexes. Computer-Instrument Interfacing: Developed a software based interface system to allow computer data aquisition and processing. Chemistry Instruction and Laboratory Supervision: Instructed and evaluated college chemistry labs and classes.

1980-1981:

Research Assistant

Chemistry Department

State University of New York at Oswego

Oswego, New York

Electrochemical Analysis: Developed analytical methods for the use of ion-selective electrodes in non-aqueous stability studies.

EDUCATION:

<u> 1987:</u>

M.S. Chemistry SUNY at Oswego Oswego, New York

<u> 1981:</u>

B.S. Chemistry
Minor: Mathematics
SUNY at Brockport
Brockport, New York

PUBLICATIONS:

Yogis, G.A., 1991. Hydrocarbon analyses: methods, case histories and comments. Presented at the 13th annual meeting of the Association of Maine Environmental Laboratories. March 26, 1991. Augusta, Maine.

Yogis, G.A., 1987. Introduction to computer interfacing: an absorbance-time data acquisition experiment. Presented at Quest Meeting, April 8, 1987. Oswego, New York.

Yogis, G.A., R.J. Scrudato and W.H. McDowell, 1986. Mirex in sediment and biota of Lake Ontario. Presented at the International Association for Great Lakes Research, 29th Conference, May 26-29, 1986, Toronto, Canada.

McDowell, W.H., R.J. Scrudato and G.A. Yogis, 1986. Mirex/photomirex ratio in Lake Ontario. Presented at the International Association for Great Lakes Research, 29th Conference, May 26-29, 1986, Toronto, Canada.

Unger, P.A., S.B. Brandt and G.A. Yogis, 1987. Acoustic assessment of fish stocks in small lakes; effects of fish and zooplankton distributions. Presented at American Fisheries Society, New York Chapter, Rome, New York.

Bixler, J.W., M. Cobb, R. French, T. O'Toole, J. Schudel and G.A. Yogis, 1987. Stability sequences of cadmium (II) and zinc (II) monohalide complexes in alcohols and binary solvents mixtures containing methanol, dimethylsulfoxide, acetonitrile and water. Inorganica Chemica Acta, 128:105-111.

SCOTT K. O'NEIL: SENIOR CHEMIST

PROFESSIONAL EXPERIENCE:

1990-Present:

Senior Chemist

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Senior Chemist involved in organics analysis; data interpretation; computer assisted chromatographic review and editing; maintain and utilize gas chromatographic instrumentation; edit, review, package final reports for customers; archive sample extracts and maintain computer files; apply expertise in PCB and Pesticide residue analysis to complex sample matrices.

1988-1990:

Senior Biochemical Technician

Health Research Inc.

Wadsworth Center for Laboratories and Research

Empire State Plaza Albany, New York 12201

Under the general supervision of a Senior Scientist, developed an experimental model and procedures for studying the effects of PCBs on the neurotransmitters and their metabolites in developing rodent brain tissue.

<u>1984-1987</u>:

Senior Chemist
Sterling Drug Inc.
East Greenbush Plant
Rensselaer, New York

Under the general supervision of the Laboratory Manager, supervised a staff of 10 chemists and 3 technicians in the performance of quality control testing on pharmaceutical raw and finished materials. Maintained responsibility for method development, training, and instrumentation operability. In addition, assisted Laboratory Manager in budget development, purchasing, and manpower efficiency studies.

1979-1983:

Analytical Chemist
Sterling Drug Inc.
East Greenbush Plant
Rensselaer, New York

Developed and implemented techniques for the quality control analysis of active ingredients in solid and liquid pharmaceutical dosage forms. Methodològies included: High performance liquid Chromatography; Gas-Liquid Chromatography; and Atomic Absorption Spectrophotometry. Conducted training for other staff in the above areas. Implemented maintenance and repair program for instrumentation used in the above areas.

<u>1975-1979</u>:

Research Technician

Department of Drug Metabolism

Sterling-Winthrop Research Institute

Rensselaer, New York

Under the general supervision of a senior researcher, developed, validated, documented, and published methodology for the microanalysis of experimental pharmaceutical compounds in human and animal body tissues and fluids.

1975-1975:

Laboratory Technician NYS Health Department New Scotland Avenue Albany, New York

Conducted hemaglutination inhibition and compliment fixation tests on various species of serum samples for encephalitis antibodies. Collected samples from wild mammals in a field environment.

EDUCATION:

<u> 1975:</u>

A.A.S. Medical Laboratory Technology Hudson Valley Community College

Troy, New York

1976-1980:

Continuing Education 20 Undergraduate Hours

PUBLICATIONS:

- F.H. Lee, R. Koss, S.K. O'Neil, M.P. Kullberg, M. McGrath and J. Edelson, Journal of Chromatography, 152 (1978) 145. "High-Performance Liquid Chromatographic Determination of Plasma and Urinary 1-Ethyl-1, 4Dihydro-4-Oxo-1, 8-Naphthyridine-3, 7 Dicarboxylic Acid."
- M.P. Kullberg, R. Koss, S.K. O'Neil, and J.Edelson, <u>Journal of Chromatography</u>, 173 (1979) 155. "High-Performance Liquid Chromatographic Analysis of Rosoxacin and its N-Oxide Metabolite in Plasma and Urine."
- G.B. Park, R.F. Koss, S.K. O'Neil, G.P. Palace, and J. Edelson, Analytical Chemistry, Vol. 53, No. 4, April 1981, 605. "Determination of Sulfinalol Hydrochloride in Human Plasma and Urine by Liquid Chromatography with Amperometric Detection."

THOMAS C. HYNES: INORGANICS LABORATORY MANAGER

PROFÈSSIONAL EXPERIENCE:

PRESENT:

Inorganics Laboratory Manager

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Responsible for the implementation of the inorganic section of Northeast Analytical that adheres to NYSDOH and EPA protocal for the preparation and analysis of environmental samples.

1990-1992:

Chemist II/Metals Supervisor CTM Analytical Laboratories

15 Century Hill Drive Latham, New York 12110

Responsible for the analysis of environmental samples through the use of ICAP, AA furnace and AA flame technique. Supervised and trained personnel in metals analysis and sample preparation.

<u>1986-1989</u>:

Chemist I/Wet Chemistry Supervisor

CTM Analytical Laboratories

Responsible for the analysis of environmental samples through the use of UV and IR Spectroscopy, gravimetry, and titration techniques. Supervised and trained personnel in wet chemistry procedures and sample preparation.

EDUCATION:

<u> 1985</u> :

B.S. Chemistry

SUNY at Plattsburgh Plattsburgh, NY

JOHN B. MILLER: ASSOCIATE CHEMIST

PROFESSIONAL EXPERIENCE:

1991-PRESENT: Associate Chemist

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Prepares samples for analysis in organics section. Dilutes final samples for packed column PCB analysis. Quantitates total PCB's by packed column method and using this data, appropriately dilutes samples for high resolution or total PCB analysis. Analyzes samples by packed column method (P60).

<u> 1989-1990:</u>

Engineering Technician/Field Services
Empire Soils Investigation, Inc.
Ballston Spa, New York

Asbestos air monitoring, steel and rebar inspections, fire proofing inspections, soil collection, handling, storage, and testing (standard and modified proctors, sieve analysis), Density/moisture soil testing, concrete cylinder testing and inspection. Customer service representative. Inspection of installation of sanitary storm and water lines.

<u>1987-1989</u>:

Environmental Chemist/Field Technician Clean Harbours, Inc. Braintree, Massachusetts

Environmental site assessments, Toxic/hazardous spill response team, QA/QC of field sampling, collection, handling, storage, transport to analytical labs. Assisted in the development, construction and maintenance of various monitoring wells, oil water separators and air strippers. Customer service representative. Familiar with, EPA methodology, OSHA regulations, Federal and State hazardous materials laws, MSDS (health and safety), and manifest and chain of custody procedures.

EDUCATION:

1985: B.S. Chemistry

B.S. Environmental Engineering Rensselaer Polytechnic Institute

1981: University of North Carolina/Chapel Hill

Major: Chemistry

CERTIFICATIONS: 40 hour Hazard Materials Training Course. Troxler Certified (Atomic/Radioactive gauge used to determine the dry density/moisture content of a pre-determined layer of soil). New York State Asbestos Handlers License

WILLIAM A. KOTAS: ASSOCIATE CHEMIST

PROFESSIONAL EXPERIENCE:

1991-PRESENT:

Associate Chemist

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Prepares samples for analysis in organics section. Reviews and compiles sample quality control information in organics sections. Implements and develops software programs for computer-local area network and oversees network operations. Performs analysis on soil and water samples for volatile organic compounds.

<u> 1990:</u>

Microscopist/Analyst

Entek Environmental & Technical Services, Inc.

Troy, New York

Analysis of air and bulk materials for the presence of asbestos fibers by Phase Contrast and Polarized Light Microscopy. Assisted in the development of a comprehensive training examination for new analysts.

<u> 1988-1990:</u>

Environmental Analyst/Quality Control Coordinator

Professional Service Industries Pittsburg Testing Lab Division

Albany, New York

Analysis of air and bulk samples for asbestos. Collection and compilation of quality control data generated by each analyst. Assistant to lab manager in daily operations and client relations.

EDUCATION:

<u> 1990:</u>

B.S. Physics
Minor: Chemistry
SUNY at Albany

Albany, New York

1985:

A.S. Mathematics/Science (Graduated with honors)

Hudson Valley Community College

INGA C. HOTALING: ASSOCIATE CHEMIST

PROFESSIONAL EXPERIENCE:

1991-PRESENT:

Associate Chemist

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Responsibilities include extraction and purification of water samples for High Resolution PCB analysis. Preparation and analysis of environmental samples for metals using Varian AA Furnace and Flame.

<u> 1989-1991</u>:

Stallion Manager Highcliff Farm

Division of Tri-Noble Stable

Delanson, New York

Provided and maintained high quality care for thoroughbred breeding stallions and surroundings. Assisted with surgeries, semen evaluation, transtrachea wash, plasma transfusion, vaccination, x-rays, and administered medication.

<u> 1986-1989:</u>

Broodmare Manager

Schoenborn Brothers Farm

Climax, New York

Responsibilities included organization of daily work schedule, mare and foal care, sales preparation, and assistance in veterinarian reproductive work.

1986:

Chemical Laboratory Assistant

Smith & Smith Environmental Laboratory

Sierra Vista, Arizona

Initiated GC procedure for testing of herbicides in soil and sludges. Completed bacteriological and chemical water analysis. Other duties included water sample collection and preparation.

<u>1383-1985</u>:

Chemical Laboratory Assistant

National Agricultural

Research & Development Institute

Augustenberg, Germany

Planned and carried out analytical procedures in connection with several research grants which included studies of heavy metals in sewage sludge and their effect in agriculture. Uptake of heavy metals by crops in relation to their concentration in the soil solution. Determination of pesticides and PCBs in municipal waste waters and the uptake of strontium-90 in agricultural crops in connection to heavy metal content and soil type.

EDUCATION:

<u> 1983:</u>

Karl-Engler School National Agricultural

Research & Development Institute

Augustenberg, Germany

MARK F. MCTAGUE: ASSOCIATE CHEMIST

PROFESSIONAL EXPERIENCE:

1991:

Associate Chemist

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Prepares samples for analysis in organics section. Performs GC analysis on water and soil samples for volatile organics. Extracts and analyzes oil and grease samples. Member of safety committee. Performs sample extracts on medium and high level PCBs. Trains prep chemists on preparation and extraction techniques.

1989:

Alcon Laboratories
Toxicology Department
2600 South Freeway
Fortworth, Texas

Assisted in preparation and execution of toxicology studies including dosing and restraining of animals and necropsy. Conceptualized and implemented a time-saving computerized archival system for slides and tissues. Developed and preformed numerous biochemical assays.

EDUCATION:

<u> 1991:</u>

B.S. Biology Minor: Chemistry SUNY at Albany Albany, New York

PROFESSIONAL EXPERIENCE:

APRIL 1992- PRESENT: Associate Chemist

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Responsibilities include extraction and purification of water samples for High Resolution PCB analysis.

<u> 1990 - 1992:</u>

Analytical Chemist

Environment One Corporation

2773 Balltown Road Schenectady, NY 12309

Perform analysis of pesticides, PCB's, herbicides, phthalates, phenols and hydrocarbons in water, oil, air and soil samples. Methods of extraction include, steam distillation, soxhlet, sonication, liquid/liquid, charcoal tube desorption. Prepare standards and samples for analysis. Removal of interfacing substances for instrumental analysis of organics. Handle data and calculations. Wet chemical analysis including titration for Nitrate, Sulfate and Cyanides, chemical oxygen demand, and total organic compounds. Use of Omega data management program. Operate electron capture GC and FID GC (Perkin Elmer), Digester, Dohrman Carbon Analyzer, and spectrophotometers. Participate in NYS DOH, NIOSH & EPA proficiency tests. Perform internal QA/QC test using surrogate spikes and standard mixtures to obtain percent recovery. Work independently and with other chemist.

<u> 1985</u>:

<u>Lab Assistant</u>

Albany Medical Center

Selected to participate in coop-education work program. Assigned to medical laboratory. Prepared tissue and blood samples for medical analysis. Prepared and filed medical records. Library searches for medical references.

EDUCATION:

1990:

B.S. Chemistry
Minor: Mathematics
SUNY at Albany
Albany, New York

TRACY M. BENGTSON: SAMPLE CUSTODIAN

1990-PRESENT:

SAMPLE CUSTODIAN

Northeast Analytical, Inc.

301 Nott Street

Schenectady, New York 12305

Sample Custodial responsibilities include sample login, storage, and tracking. The initialization, maintenance and tracking of freezer archive projects. Maintenance of sample tracking logbooks, chain of custody and manifest documentation, MSDS log, and client records. Prepares client deliverables and certificates of analysis. Prepares samples for shipment following NYS DOT Hazardous Material Protocols and shipping regulations. Client relations.

MARY STOLL:

CONTRACT LABORATORY PROTOCOL DATA PACKAGER

WENDY LEE-WAGNER:

CONTRACT LABORATORY PROTOCOL DATA PACKAGER

RON SHERMAN:

FACILITIES MANAGER